ANALYTICAL ABSTRACTS

Dealing with all branches of Analytical Chemistry: Published Monthly with THE ANALYST by the Society for Analytical Chemistry

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Published for the Society by
W. HEFFER & SONS, LTD., CAMBRIDGE, ENGLAND

Volume 5 Price 8s.
Subscription Rate, inclusive of Index, 75s. per annum, Post Free
No. 5, Abstracts 1425-1751 May, 1958

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II. Systematic errors. K. Doerffel. *Ibid.*, 1957, **157** (4), 241–248.—A procedure for the investigation of systematic errors in a series of analytical determinations is given. Knowledge of the "true" value of the single determinations is not necessary, though with small series (4 to 10 samples) the duplicate readings taken with larger series must be replaced by triplicate determinations. The system has been successfully used for several wet analytical methods and can also be used for testing new techniques. L. S. Adler
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1426 **Utilisation of complexones.** L. Domange (Fac. Pharm., Paris). *Ann. Falsif.*, 1957, **50**, 229–239; 270–281.—A review. N. E.

1427 **Qualitative analysis and analytical chemical separations without the use of sulphides.** P. W. West and M. M. Vick (Louisiana State Univ., Baton Rouge, U.S.A.). *J. Chem. Educ.*, 1957, **34**, 393–400.—Systems of qual. analysis that avoid the use of sulphides are reviewed and a new system (known as "the LSU scheme") is described. This is based on (*i*) pptn. of AgCl and Hg_2Cl_2 with HCl, followed by (*ii*) addition of sodium and ammonium benzoates at pH 4 and boiling (pptn. of H_2SnO_3, BiOCl, SbOCl and basic benzoates of Fe, Al and Cr), (*iii*) addition to the filtrate of *M* NH_4F (pptn. of fluorides of Pb, Mg, Ba, Sr and Ca), and (*iv*) addition to the filtrate of excess of aq. NH_3, and evaporation to expel excess of NH_3 (pptn. of Mn, Fe^{III}, Hg, Cu, Ni, Cd and Co). Detailed schemes for separation of each group of pptd. elements are tabulated and the basic chemistry of the scheme is discussed in detail. S.C.I. Abstr.

1428 **The use of alkali hydroxides for the separation of the copper and arsenic groups.** R. A. Chalmers and D. M. Dick (Chem. Dept., Univ. of Aberdeen, Scotland). *Analyst*, 1957, **82**, 652.—It has been recommended that Sn^{II} should be oxidised before pptn. as sulphide if alkali hydroxides are to be used for the separation of groups 2A and 2B. An oxidant that is free from the objections raised to some oxidants is $HgCl_2$, but its use entails previous detection of the Sn^{II}. If Sn^{II} is present, Hg^{II} will not be co-existent with it, and if it is absent, oxidation is unnecessary. There is thus no risk of failing to detect Hg originally present. The HgCl formed is removed, and the pptn. of group-2 sulphides is made in the usual way; HgS is found in groups 2A and 2B and is ignored. Bivalent Sn is detected by means of the cacotheline spot-test; the only common ion interfering with this test is $S_2O_3^{2-}$ in relatively large amount, but since the test is applied after removal of the group-I ppt. its presence is unlikely. A. O. Jones

1429 **The use of adsorption columns for effecting precipitation separations.** E. Hesford (A.E.R.E., Harwell, England). A.E.R.E. Report C/M 222, 1957, 6 pp.—Pptn. separation of cations without filtration (Glueckauf, A.E.R.E. Report C/R 1443, 1954) can also be effected by passing the sample soln. through a column (1 in. diam.) of activated carbon (30 to 40 mesh) pre-treated with a 5 to 10% aq. soln. of an organic amine, and then removing one or more of the washed pptd. cations with aq. acid or weak organic base. Thus, Fe can be separated completely from Al by passing a soln. containing 0·5% of $FeCl_3$ and 0·05% of $Al_2(SO_4)_3$ through the column pre-treated with triethanolamine, Al being retained on the carbon; Ni and Co can be separated by passing the soln. containing 0·2% of $CoSO_4$ and 0·2% of $NiSO_4$ through the column pre-treated with ethanolamine and then removing the pptd. Ni by elution with a 5% aq. soln. of the same base. Other separations (Cu from Cd and U from Th or other cations) can be made by reference to the pptn. pH of cations, the dissociation const. of water-sol. bases, and some observed pptn. reactions (listed). W. J. Baker

1430 **A series of fluorescent acid – base indicators.** G. N. Kosheleva. Report of the Conference on "Luminescence analysis," Minsk, Akad. Nauk BSSR, 1956, 31–36; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,803.—The following series of acid-base indicators, which give a blue-green fluorescence, is recommended (the pH range of the fluorescence is given in brackets)—3-ethoxyacridine (1·3 to 3·2), 2-naphthylamine (2·8 to 4·4), 1-naphthylamine (3·4 to 4·8), acridine (4·8 to 6·6), β-methylumbelliferone (5·8 to 7·5), G-salt (7·5 to 9·0), R-salt (8·0 to 10·5), FF-acid (8-amino-1-naphthol-5:7-disulphonic acid) (10·0 to 12·0) and 1-naphthylamine-5-sulphonic acid (12·0 to 13·0). C. D. Kopkin

1431 **Application of amperometric dead-stop end-point to iodate procedures in the presence of mercuric mercury.** M. G. Bapat and B. Sharma (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **157** (4), 258–261 (in English).—Arsenous oxide and hydrazine sulphate are used as direct primary standards, and thiosulphate, thiocyanate and thiourea are determined by using an excess of iodate and back-titration. E. G. Cummins

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1432. **The EDTA titration: nature and methods of end-point detection. III.** A. J. Barnard, jun., W. C. Broad and H. Flaschka (Baker Chemical Co., Phillipsburg, N.J., U.S.A.). *Chemist Analyst*, 1957, **46** (2), 46–54.—The indicators used in EDTA titrations are reviewed and the methods of end-point detection by fluorescence, turbidity and redox effects are discussed. D. P. FELIX

1433. **Paper chromatography and paper electrophoresis.** [I]. A. Grüne (C. Schleicher & Schüll, Dassel). *Chimia*, 1957, **11** (7), 173–203.—A review, with 49 references. A. B. DENSHAM

1434. **Experimental precision in quantitative analysis by gas chromatography.** R. Maurel (Lab. Chim. de l'École Normale Supérieure, 24 rue Lhomond, Paris). *Compt. Rend.*, 1957, **244** (26), 3157–3159.—A study of the quant. analysis, by gas chromatography, of mixtures of 1:1:3-trimethyl*cyclo*hexane, *m*-xylene and *pseudo*cumene, with a thermal conductivity cell as detector, shows that, since the millivoltmeter does not exactly follow the variations of p.d. at the peaks, a correction factor is necessary. S.C.I. ABSTR.

1435. **Proceedings of the Colloquium Spectroscopicum Internationale VI. Amsterdam. May 1956.** *Spectrochim. Acta*, Special number, 1957, 663 pp.—The following papers of analytical interest are included (in German unless otherwise stated). **The mutual influence of the emission of sodium and potassium in their flame-photometric determination,** K. E. Stumpf and T. Gonsior, 35–43. **Determination of sodium by flame spectrophotometry in aluminium oxide,** L. Braicovich and M. F. Landi, 51–61 (in French). **The indirect flame-photometric determination of aluminium,** J. Cencelj, 62–66. **The quantitative spectral analysis of small amounts of caesium and rubidium,** J. Medek and F. Valeška, 67–70. **Advantages of a calculating device in direct-reading spectral analysis,** C. Burkhardt, 84–88. **The introduction of a photo-electric instrument for serial micro-analysis in the investigation of structural constituents of steel,** W. Koch and S. Eckhard, 89–93. **The application of short-wave u.v. lines for analysis by direct measurement,** G. Limmer, 94–96. **The complementary nature of X-ray fluorescence methods and optical emission methods in spectrochemical analysis,** M. F. Hasler, 97–104 (in English). **Trends in automatic spectrochemical analysis,** A. C. Menzies, 106–110 (in English). **The background in the spectrum of the carbon arc and its significance in spectral analysis,** E. Golling, 125–133. **"Enhancement" and "weakening" in spectrochemical analysis. Effects of the source energy and the excitation potential of the lines of the elements. Analytical deductions,** V. Gazzi, 134–139 (in French). **Volatilisation phenomena in the direct current arc used as a spectrochemical light source for the analysis of powders,** J. Beintema, 186–191 (in English). **Stabilisation of the vaporisation of the filled cathode in a d.c. carbon arc,** M. F. A. Hoens and J. A. Smit, 192–196 (in English). **A comparison of a.c. arcs of different voltages,** K. Laqua, 197–204. **Effect of micro-structure of the specimen on the spectrochemical analysis of steels,** Ho I-djen and Chang Kung-soo, 205–211 (in English). **Spectrographic analysis of slags by the cup-electrode solution arc method,** Ho I-djen, Li Shih-cho and Wang Thang-soo, 212–216 (in English). **Correlation between the processes on the electrodes and the spectral emission,** Z. Šternberg, 217–221 (in English). **The "sliding" spark in a vacuum as a source for emission spectrochemical analysis,** J. Romand, G. Balloffet and B. Vodar, 268–274 (in French). **A composite procedure for the spectrographic determination of impurity and alloying elements in steel,** R. H. Tyas, 275–281 (in English). **The influence of the electrical parameters of an intermittent d.c. arc unit on the distribution of spectral energy across the analytical gap,** K. M. Bills, 282–288 (in English). **Spectrochemical analysis of grey cast-iron,** H. Moritz, 291–297. **Spectrographic determination of carbon in low- and high-alloy steels,** J. Bruch, 298–305. **Spectrographic determination of carbon in steels,** T. Török and G. Szikora, 306–309. **Spectrochemical determination of selenium in pyrites,** E. Schroll and W. Rockenbauer, 338–341. **Production of spark lines in a hollow cathode tube with pulse emission,** M. Becart, G. Deprez and J. Roig, 342–349 (in French). **New monochromatic interference filters,** R. Schläfer, 361–367. **A triggered 220-V a.c. spark for high precision in metal analysis,** C. G. Carlsson, 370–376 (in English). **Spectrochemical trace analysis after previous concentration,** K. Scharrer and G. K. Judel, 377–381. **A method for increasing the sensitivity in the spectrochemical analysis of solutions with carbon electrodes,** F. J. Haftka, 382–385. **The "equal-intensity" procedure; a routine method for general spectrochemical analysis,** W. Seidel and H. J. Eichhoff, 409–419. **Comparison of arc- and spark-excitation in a special method of steel analysis,** A. Camuñas and H. Carrancio, 437–440 (in French). **Quantitative spectral analysis of traces of contaminating elements in spectrographic carbon in the d.c. continuous arc,** F. Hegemann and H.-H. Rüssmann, 448–456. **Design and performance of a fore-prism-grating infra-red spectrometer,** E. H. Siegler, jun., and J. W. Huley, 503–506 (in English). **Fluorescence spectra of "burning" kerosines,** C. Mihul, C. Ruscior and V. Pop, 562–564 (in French). **The identification and quantitative determination of tertiary butyl groups in hydrocarbons by Raman and infra-red spectroscopic methods,** W. Boog and E. D. Kunst, 568–572 (in English). **Infra-red spectra of phthalocyanines with different central metal atoms,** A. N. Terenin and A. N. Sidorov, 573–578 (in English). **Infra-red study of some organic salts in solution,** C. la Lau and E. A. M. F. Dahmen, 594–600 (in English). **Infra-red spectra of some organic sulphur - oxygen compounds,** S. Detoni and D. Hadzi, 601–602 (in English). **Absorption spectra of H_2O - D_2O mixtures in the liquid state between 0·8 μ and 30 μ,** M. Ceccaldi, M. Goldman and E. Roth, 623–631 (in French).

1436. **High-precision spectrophotometric analysis.** M. Q. Freeland and J. S. Fritz (Iowa State Coll., Ames). *U.S. Atomic Energy Comm.*, Rep. ISC-667, 1956, 31 pp.—Procedures are presented in which a modification of the Beer's law equation is used that permits the use of unmatched cuvettes for high-precision spectrophotometric analysis. The errors due to the use of unmatched cuvettes are calculated, and they indicate the optimum conditions relative to mismatching, for the use of the existing methods. These procedures were applied to the analysis for V in three V - Al alloys. The accuracy attained on synthetic samples averaged 0·1%; the reproducibility on the actual samples averaged better than 0·2%. CHEM. ABSTR.

1437. **The exact consideration of the blank value in colorimetric determinations.** J. Agterdenbos (Lab. Anal. Chem., Univ. Amsterdam, Holland). *Z. anal. Chem.*, 1957, **157** (3), 161–165.—The accuracies of various ways of considering the blank

extinctions are compared. It is shown that the methods where the test soln. is measured directly against the blank soln. are always more accurate than those where a single separately determined blank value is subtracted from the measured values of successive test soln. If the mean of two or more measured blank values is subtracted, greater accuracy is possible in some, but not all, cases than if a single value is subtracted. A method recommended by Sandell is in general more accurate than that of the subtraction of a mean blank value. It is seldom more accurate than direct measurement of test soln. against blank. L. S. ADLER

1438. **New trends in polarography.** J. Heyrovský (Polarographic Inst., Czech. Acad. Sci., Prague). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 3–16.—A review. G. H. FOXLEY

1439. **Polarographic analysis with alternating current.** B. Ya. Kaplan (Chem. Lab. of the Geological Dept. of the Central Regions). *Zavod. Lab.*, 1957, **23** (7), 771–780.—A review with 45 references, together with a description of a new "pulse-polarograph," is presented. G. S. SMITH

1440. **Principles of isotope dilution assays.** C. Rosenblum (Merck & Co., Inc., Rahway, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1740–1744.—The basic principles of isotope dilution, reverse isotope dilution, multiple dilution, isotope derivative and derivative dilution are described and the conditions for reliable assays are enumerated. The stability of vitamin B_{12} in dry cereals at concn. as low as 0·46 μg per oz has been demonstrated by the reverse isotope dilution procedure. K. A. PROCTOR

1441. **Neutron activation analysis.** M. J. Cabell. *Ind. Chem. Mfr*, 1957, **33**, 345–351.—The principles, experimental methods in use, sensitivity and limitations of the neutron activation technique are reviewed and some of its applications described. (29 references.) S.C.I. ABSTR.

See also Abstracts—**1442**, Analytical properties of benzil and anisil dioximes. **1661**, Chronometric micro-analysis.

2.—INORGANIC ANALYSIS

1442. **Report of the Conference on Methods of Analysis of Rare and Non-Ferrous Metals.** Moscow, MGU, 1956. The following papers read at the Conference have been abstracted in *Ref. Zhur., Khim.*, 1957, (13). The page numbers of the report and the abstract numbers are given after each title. **Study of the analytical properties of benzil and anisil dioximes,** V. M. Peshkova and I. I. Nazarenko, 3–14; 44,812. **Microchemical determination of copper with bromobenzotriazole,** L. N. Lomakina and N. I. Tarasevich, 33–39; 44,844. **Potentiometric determination of lead and barium ions when both are present,** P. K. Agasyan, 41–45; 44,825. **Colorimetric determination of germanium as the molybdogermanic heteropoly acid,** Z. F. Shakhova and R. K. Motorkina, 47–57; 44,856. **Separation of ter- and sexa-valent chromium by ion-exchange chromatography,** T. A. Belyavskaya and E. P. Shkrobot, 59–62; 44,867. **Spectrographic determination of iron, titanium and tin impurities in tantalum pentoxide,** N. I. Tarasevich and L. V. Shuvalova, 63–70; 44,890. **Qualitative reactions for caesium (rubidium, ammonium, potassium), cadmium and copper,** A. S. Kozlov, 71–78; 44,841. **Reaction of nickel and copper with dimethylglyoxime in the presence of oxidising agents,** V. M. Peshkova and N. V. Mel'chakova, 93–103; 44,813. **Separation of niobium and tantalum with acridine,** I. M. Gibalo and K. L. Malyarov, 105–108; 44,817. **Removal of titanium from niobium and tantalum with acridine,** K. L. Malyarov and I. M. Gibalo, 109–115; 44,818. C. D. KOPKIN

1443. **The use of amperometric titrations in the analysis of metals.** I. Bozsai (Mat. Testing Lab., Csepel Iron and Metal Works, Budapest). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 195–222.—The amperometric titration of Cr, V and Mn (as $Cr_2O_7^{2-}$, VO_3^- and MnO_4^-, respectively) in steel is described; rotating platinum and calomel electrodes are used. Antimony, As, Bi and Te are determined (as Sb^{3+}, As^{3+}, Bi^{3+} and TeO_3^{2-}, respectively) by oxidation with $K_2Cr_2O_7$ and back-titration with Fe^{2+}. Bivalent Co is oxidised to Co^{3+} in ammoniacal tartrate soln. by $[Fe(CN)_6]^{3-}$, and the excess of this reagent determined by titration with Co^{2+}. Direct titration with $[Fe(CN)_6]^{4-}$ is suitable for the determination of Zn^{2+}, and Ag^+ may be titrated with Cl^- or Br^-. Copper, Ni, Zn, Co^{3+}, Co^{3+}, Al and Fe in CO_2-free soln. can be titrated with 0·1 *N* NaOH. G. H. FOXLEY

1444. **The determination, by means of bromocyanogen, of cyano-complex-forming metals.** E. Schulek, E. Kőrös and J. Kéthelyi (Inst. of Inorg. and Anal. Chem., Eötvös Loránd Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1957, **63** (8), 229–232.—The use of this iodimetric method (*Mikrochem. Sonderband Emich-Festschrift*, 1930, 260) is examined for the determination of some other metals. The cyano complexes of Cd, Zn and Ni are completely decomposed by boiling with boric acid, but the Cu^{II} complex loses a variable amount of cyanide. Formaldehyde decomposes the complexes of Cd and Zn completely and those of Cu^{II} and Ni only partially. By measuring the cyanide, Cu and Ni can be determined with an accuracy up to 2%. A. G. PETO

1445. **Use of reactions for the synthesis of certain organic dyes in the qualitative analysis of anionic oxidants.** S. S. Vil'borg and V. A. Drozdov. *Trudy Moskov. Khim.-Tekhnol. Inst.*, 1956, (23), 130–134; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,811.—A study is made of the oxidation reactions of tannin (to form the dye ellagic acid). For detecting the oxidants ClO_3^-, BrO_3^-, IO_3^-, ClO_4^-, $S_2O_8^{2-}$, NO_2^-, NO_3^-, AsO_4^{3-} and H_2O_2, 1 ml of a 3% soln. of tannin is added to 1 ml of a soln. of the oxidant and 5 or 6 drops of acetic acid (to increase sensitivity). It is established that there is dye formation when tannin is oxidised with IO_3^-, NO_2^- or $S_2O_8^{2-}$, with sensitivities 17, 21 and 120 μg per ml, respectively. Similar reactions of *o*-toluidine (**I**) and *m*-tolylenediamine (**II**) with the same oxidants are also examined. In the cold, in the absence of free acid, **I** does not form a dye with $S_2O_8^{2-}$, ClO_4^-, ClO_3^-, BrO_3^-, IO_3^-, AsO_4^{3-} or H_2O_2. A positive reaction is given by **I** and **II** with ferricyanides (sensitivity 27 μg per ml) and by **II** with persulphates (25 μg per ml). No dye is formed by **II** with ClO_2^-, BrO_3^-, IO_3^-, ClO_4^- or AsO_4^{3-}. C. D. KOPKIN

1446. **Radio-assay of low specific-activity tritiated water by improved liquid scintillation techniques.** C. A. Ziegler, D. J. Chleck and J. Brinkerhoff (Tracerlab, Inc., Waltham, Mass., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1774–1776.—The performance of the liquid scintillation method can be considerably improved by making the ratio of water sample to scintillation soln. optimum and by de-oxygenating the scintillation soln. by ultrasonic de-gassing. The technique can be applied to the radio-assay of other water-soluble radio-isotopes.

K. A. PROCTOR

1447. **Complexometric control of the quality of distilled water.** O. Yu. Kaleis (Control Analytical Lab., "GAPU," Latvian SSR). *Aptechnoe Delo*, 1957, **6** (2), 29–32.—By titrating the sample against Trilon B (EDTA), as in the determination of hardness, 100 ml of water should give a titre of ≥ 1 ml of 0·01 *N* EDTA. E. HAYES

1448. **Spectrographic determination of lithium in copper.** T. K. Aidarov. *Trudy Vses. Nauch. Inst. Galurgii*, 1956, (31), 188–190; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,677.—Dissolve the sample (8 g) in conc. HNO_3 (40 ml), make up to 100 ml and introduce two or three drops on to the hot lower carbon electrode, which has a crater of 3 mm diam. and depth 2 mm. Excite the spectrum in an a.c. arc discharge with inter-electrode gap of 2 mm and current strength 4·5 amp. Compare the analytical line at 6707·8 A with the background near the line. Prepare standards from soln. of pure Cu containing 0·001 to 0·05% of Li. Construct the calibration curve with the co-ordinates ΔS and $\log C$. The error of the analysis is $\pm$ 10%.

C. D. KOPKIN

1449. **The determination of sodium and potassium in a sample of dunite by radioactivation analysis.** L. Salmon (A.E.R.E., Harwell, England). A.E.R.E. Report C/M 323, 1957, 6 pp.—The radioactivation of dunite and the radiochemical separation methods for the determination of K and Na are described. Details are given of the use of the gamma spectra illustrated to check the values for Na. On four determinations the average values were K = 0·0032%, Na = 0·011%.

E. G. CUMMINS

1450. **Determination of potassium by the dead-stop method. Sodium tetraphenylboron as precipitant and thallous nitrate as volumetric reagent.** H.-J. Schmidt (Chem. Fabrik Kalk G.m.b.H., Cologne, Germany). *Z. anal. Chem.*, 1957, **157** (5), 321–338.—A method is described for the determination of potassium, ammonium and salts of organic bases, which is based on the pptn. of these substances with sodium tetraphenylboron soln. and back-titration with $TlNO_3$ soln. The details of electronics incorporated in the construction of the instrument used for end-point determination are given. E. G. CUMMINS

1451. **Flame-photometric analysis of minerals for alkali-metal content.** A. Willgallis (Mineralog. Inst., Freien Univ., Berlin). *Z. anal. Chem.*, 1957, **157** (4), 249–257.—Details are given of an atomising burner and the influence of interfering elements (Li, Ca, Mg, Al, Fe) is discussed. Notes on the decomposition of the samples and the measuring system are included. A relatively low flame temp. is used. E. G. CUMMINS

1452. **Photocolorimetric determination of small amounts of copper by means of 2:2′-diquinolyl [cuproine].** A. L. Gershuns and Yu. V. Bashkevich (Sci. Res. Inst. of Chem., A.M. Gorki Kharkhov State Univ.). *Zavod. Lab.*, 1957, **23** (7), 787–788.—The cuproine method is applied to the determination of small amounts (0·0015 to 0·05%) of Cu in aluminium and zinc. G. S. SMITH

1453. **Determination of copper(II) ions by means of ascorbic acid.** L. Erdey and G. Siposs (Inst. Allg. Chem., Tech. Univ., Budapest). *Z. anal. Chem.*, 1957, **157** (3), 166–177.—Several methods of the direct reductimetric titration of Cu^{2+} with standard ascorbic acid soln. have been investigated. The various media in which the titrations could be carried out are (*i*) $Fe(NH_4)_2(SO_4)_2$ - KSCN, (*ii*) KBr - Na acetate, (*iii*) NaCl - Na acetate and (*iv*) NH_4Cl - Na acetate. The last named is the most suitable for practical analytical purposes. The titration is carried out in an atmosphere of CO_2 to prevent autoxidation of the ascorbic acid. The indicator is 2:6-dichlorophenolindophenol (0·1% aq. soln.). Accuracy and reproducibility are comparable with those achieved by iodimetric methods. Few other ions interfere. All reagents are cheap and the titration may be carried out both visually and potentiometrically on the macro and on the micro scale. L. S. ADLER

1454. **Study of the co-precipitation of traces of copper with the 8-hydroxyquinolinates of certain metals.** A. U. Mamenko and N. T. Chuiko. *Nauk. Zap. Cherkas'k. Derzh. Ped. Inst.*, 1956, **8**, 109–112; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,637.—In the pptn. of 0·5 millimole of Pb from 100 to 700 ml of a soln. containing 10 μg of Cu and 12 millimoles of Na acetate with excess (1·5 millimoles) of 8-hydroxyquinoline sulphate, the amount of Pb pptd. decreases with increase of the vol. of the soln. (from 76% with 100 ml to 52% with 500 ml); the amount of Cu in the ppt. decreases in the same way. Under analogous conditions and on increasing the vol. to 1 or 2 litres, the 8-hydroxyquinolates of Ni and Zn are practically completely pptd., and Cu goes quant. into the ppt. On the basis of the results of the experiments, it is suggested that in certain conditions Cu passes into the ppt. by co-pptn. as a result of adsorption of copper ions or atoms by the ppt., and not because of actual co-pptn., in spite of the fact that the concn. of Cu exceeds the value of the solubility of Cu hydroxyquinolinate, calculated from its solubility product ($4·2 \times 10^{-24}$). As a carrier in such cases, the least soluble of the metal hydroxyquinolinates must be used.

C. D. KOPKIN

1455. **Ferrometric determination of silver ions.** L. Erdey and K. Vigh (Inst. Allg. Chem., Tech. Univ., Budapest). *Z. anal. Chem.*, 1957, **157** (3), 184–192.—The method depends on the quantitative reduction of Ag^+ to metallic Ag by means of $FeSO_4$ soln. in the presence of NaF. Variamine blue is used as indicator. The reproducibility and accuracy are good; the coeff. of variation is $\pm$ 0·12%. Interference by other ions is discussed. The method is well suited for the determination of the silver content of Ag - Cu alloys. Separation of the Ag from the Cu is not necessary. L. S. ADLER

1456. **Flame-spectrophotometric determination of microgram quantities of magnesium.** L. Manna, D. H. Strunk and S. L. Adams (Joseph E. Seagram & Sons, Inc., Louisville, Ky., U.S.A.). *Anal. Chem.*,

1957, **29** (12), 1885–1887.—Experiments on the effect of organic solvents and anion and cation interference are detailed. These tests led to the selection of a radiation buffer comprising Ca (750 μg per ml), Al (25 μg per ml), and 2 *M* acetic acid, to be used with an 80% acetone soln. of the magnesium. Without the buffer, concn. of 2, 3 and 4 μg of Mg per ml could be accurately determined in the presence of 40 μg each of Al, Ca, Co, Cu, Fe, Li, K, Ni, Na and Pb. At concn. of 1, 5 and 6 μg of Mg per ml in similar experiments, the buffer was necessary to get results within 0·1 μg per ml of the theoretical value. E. G. CUMMINS

1457. **Polarographic determination of magnesium.** Z. Ferenczy, A. Almásy and G. Szádeczky-Kardoss (Res. Inst. of the Heavy Chem. Ind., Veszprém, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 179–184.—Two methods are described. (*i*) The hexamine complex of magnesium iodide is pptd., filtered off, washed, and dissolved in 0·1 *N* KNO_3 and the iodide concn. is determined polarographically. The error on 0·5 to 2·0 mg of Mg was −3%. (*ii*) The hexamine complex of calcium magnesium ferrocyanide is pptd., filtered off, washed, and dissolved in KNO_3 soln. The ferrocyanide is titrated polarographically with 0·1 *N* $Pb(NO_3)_2$. The error on 0·5 to 2·0 mg of Mg was −3% to +2%. Both methods are suitable for the analysis of silicates. G. H. FOXLEY

1458. **Complexometric determination of magnesium in aluminium alloys.** L. M. Budanova and R. S. Volodarskaya. *Zavod. Lab.*, 1957, **23** (7), 797.—After separation of Zn and Al by means of NaOH and of Cu, Ni, Mn and Fe by means of Na diethyldithiocarbamate, Mg is titrated with 0·05 *N* EDTA (disodium salt). The sample (1 g or, with a content of Mg >3%, 0·5 g) is dissolved in 30 ml of 20% NaQH soln., 200 ml of hot water is added, the insoluble matter is filtered off and washed with hot 2% NaOH soln. and then dissolved in 30 ml of an acid mixture [300 ml of dil. HCl (1:2) and 10 ml of dil. HNO_3(1:1)]. The soln. is filtered, the filtrate is neutralised to Congo red paper with 20% NaOH soln., 80 ml of 5% Na diethyldithiocarbamate soln. is added slowly, the soln. is diluted to 250 ml and an aliquot portion is filtered off. The soln. (≃100 ml) at 40° to 50° is mixed with 3 drops of a 1% soln. of an indicator (acid chrome dark blue) and 5 to 6 drops of aq. NH_3 and titrated with EDTA. A determination can be carried out in 60 to 90 min. G. S. SMITH

1459. **A new and rapid method for determining unhydrated magnesia in dolomitic lime hydrates.** E. Trattner (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *Bull. A.S.T.M.*, 1957, (225), 53–57.—The procedure described is based on the forced hydration of the non-hydrated MgO in the sample (2 to 3 g) by steam at 295 lb per sq. in. for 1 hr. in an autoclave. On the assumption that the CaO is initially completely hydrated, only the free MgO is responsible for the gain of H_2O after treatment. This gain is determined by drying and weighing the sample, before and after hydration, to the same degree of dryness. Carbonation must be rigorously avoided throughout the analysis. Although the mean theoretical error caused by the assumptions made is +0·41%, the method is claimed to be simpler and more precise than lengthy chemical analysis (ASTM method C25–47), for which the mean theoretical error is + 0·15% on 12 limes. The standard deviations for the two methods are ± 0·10 and ± 0·50, respectively (6 to 12 analyses). W. J. BAKER

1460. **Spectrographic determination of the oxides of calcium and magnesium in clays.** B. S. Gorovaya and T. K. Aĭdarov. *Trudy Vses. Nauch. Inst. Galurgii*, 1956, (31), 191–194; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,686.—Grind the sample (0·5 g), dried to const. wt.; add water to form a suspension, spread a thin layer on a copper plate and dry. This plate serves as the lower electrode, and moves at a speed of 20 mm per min. relative to the upper copper electrode, which is sharpened to a truncated cone. Photograph the spectra on a medium spectrograph, with exposure 2 min. and arc gap 1·5 mm. The excitation of the spectra is achieved with an a.c. arc discharge. The analytical lines used are (in A) Ca 3933·7 and Cu 3317·2, and Mg 2795·5 and Cu 2700·9. Chemically analysed clays, containing 0·34 to 1·17% of CaO and 0·20 to 1·1% of MgO, serve as standards. Construct the calibration curve with the co-ordinates ΔS and log *C*. The error is ±4·5% for MgO and ±4·8% for CaO. The results agree well with chemical data and the method is 10 to 12 times as rapid as the chemical procedure. C. D. KOPKIN

1461. **Separation of strontium from calcium with potassium rhodizonate. Application to radiochemistry.** H. V. Weiss and W. H. Shipman (U.S. Naval Radiolog. Defense Lab., San Francisco 24, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1764–1766.—The hazards of the conventional 75% HNO_3 procedure are overcome by the two simple methods outlined. The extent of recovery is a function of the molar ratios of the Ca, Sr and rhodizonate. A single operation can separate 50 mg of Sr from 11 g of Ca with an 80% recovery of Sr, and a 99% removal of Ca; alternatively, 50 mg of Sr can be separated from 40 g of Ca with a 60% recovery of Sr and a 99·8% removal of Ca. E. G. CUMMINS

1462. **Rapid estimation of lithopone. I. Complexometric estimation of zinc and barium in admixture.** M. R. Verma and V. M. Bhuchar (National Physical Lab. of India, New Delhi). *Paint Manuf.*, 1957, **27** (10), 384–385, 389.—The sample soln. is adjusted to a pH of 6·7 (Na maleate - maleic acid buffer), and titrated with 0·05 *M* EDTA (disodium salt) (Eriochrome black T indicator) to give the content of Zn; the pH is then adjusted to 10 (NH_4Cl - aq. NH_3 buffer) and the titration is continued to give the content of Ba. A better end-point in the latter titration is obtained if an excess of EDTA soln. is added and back-titrated with $MgCl_2$ soln. D. R. DUNCAN

1463. **Modified iodimetric method for the estimation of zinc.** R. K. Nanda and B. Prasad (Mayurbhanj Chem. Lab., Ravenshaw Coll., Cuttack, India). *J. Indian Chem. Soc.*, 1957, **34** (6), 479–480.—*Procedure*—A soln. containing Zn (30 to 150 mg) is placed in a 500-ml stoppered flask, and KI (3 g), $(NH_4)_2SO_4$ (5 to 10 g) and *M* ammonium acetate (50 ml) are added. The pH is adjusted to between 3 and 4 with *N* H_2SO_4 (≃45 ml) and the vol. is made up to 250 ml. Enough 0·1 *M* $K_3Fe(CN)_6$ is added to give an excess of >2 millimoles per litre, and, after being set aside in the dark for 5 min., the soln. is titrated against $Na_2S_2O_3$ soln. The accuracy is within ±0·3%. C. A. SLATER

1464. **Complete polarographic analysis of cyanide zinc-plating baths using a simplified apparatus.** T. H. Collard, jun., and D. K. H. Liu (Friedrich Refrigerators, Inc., San Antonio, Tex.). *Tech. Proc. Amer. Electroplaters' Soc.*, **43***rd Ann. Conv., Washington*, 1956, 56–59.—The polarographic circuit has been modified and made simpler and less expensive. Determinations were made with it for Zn, total NaCN, NaOH and Na_2CO_3. Preliminary treatment of the original soln. is unnecessary, except that EDTA is added as a chelating agent before the determination of total NaCN, thereby reducing analytical time. CHEM. ABSTR.

1465. **Polarographic determination of cadmium and cyanide in alkaline cadmium-plating baths.** G. T. Forsyth (Udylite Res. Corp., Van Dyke, Mich.). *Tech. Proc. Amer. Electroplaters' Soc.*, **43***rd Ann. Conv., Washington*, 1956, 91–93.—Both Cd and total CN^- can be determined in 10 to 15 min. by simply diluting the plating soln., adding a supporting electrolyte and measuring the diffusion currents of Cd and CN^-. CHEM. ABSTR.

1466. **Influence of tartrates and citrates on the gravimetric determination of cadmium by the carbonate method.** G. I. Popov. *Trudy Dagest. Sovietskh. S.-Kh. Inst.*, 1956, **9**, 169–175; *Ref. Zhur., Khim.*, 1957, Abstr. No. 48,283.—The influence of tartrates and citrates on the determination of Cd^{2+} by the carbonate method is studied; it is established that in alkaline medium (pH 8 to 9·8) Na tartrate holds in soln. a considerable quantity of Cd (as $NaCdC_4H_3O_6$), this quantity increasing with increase of tartrate. Complex tartrates of Cd are not very stable; on dilution with water their soln. turn cloudy and, after a time, ppt. form slowly. In slightly acid soln. (pH 6·5 to 7) in the absence of excess of tartrate, a part of the Cd is pptd.; with excess of tartrate, Cd passes into solution; addition of K_2CO_3 favours the formation of a tartrate complex; excess of K_2CO_3 causes pptn. of part of the Cd. Sodium citrate masks Cd much more strongly than does the tartrate; at pH 8 to 9 in the presence of excess of citrate there is formed $Na_2CdC_6H_4O_7$. In a weakly acid medium with the ratio of $CdSO_4$ to citrate between 1:1 and 1:2, the main product is normal Cd citrate; on adding excess of citrate the normal Cd citrate is converted into a complex. The addition of K_2CO_3 accelerates the formation of the internally complexed $K_2CdC_6H_4O_7$. In the presence of a sufficient excess of citrate, the complete transformation of $CdCO_3$ into a complex takes place much more easily than in the presence of tartrate.

C. D. KOPKIN

1467. **Polarographic determination of boric acid by means of sodium nitrite.** H. I. Shalgosky (U.K. Atomic Energy Authority, Woolwich Outstation, London). *Analyst*, 1957, **82**, 648–649.—It has been shown that H_3BO_3 in the presence of Na_2SO_3 and mannitol gives rise to a polarographic wave that may be used for its determination (Lewis, *Analyst*, 1956, **81**, 531). It is now shown that the substitution of $NaNO_2$ for Na_2SO_3 increases the sensitivity of the method. The base electrolyte is de-oxygenated, the de-oxygenated sample soln. is added, and after 10 min. the polarogram is recorded over the range 0 to −1·2 V with a mercury-pool anode. The step height occurring at $E_{\frac{1}{2}} = \simeq -0{\cdot}7$ V is measured by the intersection method, and the concn. of H_3BO_3 is ascertained from a calibration graph prepared similarly with standard H_3BO_3 soln. Compared with the original method (*loc. cit.*) this method is three times as sensitive at 200 μg of boron per ml and ≃ 40 times at 20 μg per ml.

A. O. JONES

1468. **Polarographic determination of gallium.** V. I. Lȳsenko and P. P. Tsȳb (All-Union Sci. Res. Min. Metall. Inst.). *Zavod. Lab.*, 1957, **23** (7), 794–796.—In 2·6 *N* aq. NH_3 containing NH_4Cl or $(NH_4)_2SO_4$, Ga can be determined polarographically by means of its wave with $E_{\frac{1}{2}} = -1{\cdot}60$ to $-1{\cdot}62$ V *vs.* the S.C.E. if O is removed. When a soln. containing Na_2SO_3 and gelatin is used to remove O, the wave height is considerably less than when H is used, but it is suitable when the concn. of Ga is >10 mg per litre. Prior extraction of Ga with ether is necessary when certain elements are present. In this case, after evaporation of the ether from the extract, the residue is evaporated with a few drops of H_2O_2, 6 ml of dil. HCl (1:1) is added, the soln. is neutralised with aq. NH_3, 5 ml of 25% aq. NH_3 is added and the soln. is diluted to 25 ml. In the presence of much Fe, the Ga and some of the Fe is extracted with ether, the Ga is transferred to an aq. layer, Fe^{3+} are reduced to Fe^{2+}, and Ga is again extracted.

G. S. SMITH

1469. **Polarographic investigation of gallium.** J. Szonntagh and J. Rozmanith (Inst. of Anal. Chem., Univ. of the Chem. Ind., Veszprém, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 99–104.—Earlier difficulties in the determination of Ga were due mainly to the nearness of the hydrogen wave. It has been found that the wave height of Ga is constant in the pH range 5·35 to 6·35. Within this range the hydrogen wave does not interfere and results are reproducible.

G. H. FOXLEY

1470. **Adsorption and solvent-extraction procedures for the separation of carrier-free indium from cadmium.** L. Lawson and M. Kahn (Dept. of Chem., Univ. of New Mexico, Albuquerque). *J. Inorg. Nuclear Chem.*, 1957, **5** (1), 87–92.—The maximum adsorption of ^{115}In on powdered Pyrex glass (0·3 g) or paper mash occurred from soln. 2 to 5·3 *M* in aq. NH_3 and ≻0·01 *M* in Cd; glass was more effective than paper and at least 96% of the ^{115}In was adsorbed. Adsorption on fritted-glass discs was 30 to 99% according to flow rate and was independent of concn. of Cd up to 0·01 *M*. The adsorption on Millipore filters was 15 to 30% but was 4% from 0·01 *M* cadmium soln. The adsorption on Whatman papers was 4%. Quantitative extraction of ^{115}In into 0·01 *M* 8-hydroxyquinoline in chloroform occurs in the pH range 3·4 to 6·4 and is independent of cadmium concn. up to 0·1 *M*.

G. J. HUNTER

1471. **Vanadametry. Determination of thallium(I).** K. Bhaskara Rao, G. Jagga Rao and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1957, **157** (3), 181–183 (in English).—Univalent Tl is quantitatively oxidised to Tl^{III} by titration with standard sodium vanadate soln. in strongly acid (HCl) medium, with iodine chloride as catalyst. The titration is carried out in the presence of CCl_4, and the flask must be vigorously shaken. The reaction is slow near the end-point, which is indicated by colour change of the CCl_4 layer from purple to colourless. The HCl concn. at the end-point must be at least 7·5 *N*.

L. S. ADLER

1472. **Electrometric end-points in the argentimetric determination of cerium.** B. R. Sant (Hindu Univ., Benares, India). *Rec. Trav. Chim. Pays-Bas*, 1957, **76** (7), 590–592.—Quadrivalent Ce is determined volumetrically by reduction to Ce^{III} with excess of KSCN and electrometric titration of the excess with $AgNO_3$ soln. It is shown that potentiometric end-points in the KSCN - $AgNO_3$ reaction in the presence of sufficiently large concn. of H_2SO_4 and/or Ce^{III} yield accurate results. M. DAVIS

1473. **Electrometric end-points in the oxidation of thiocyanate by permanganate. Determination of cerium.** M. K. Joshi (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **157** (4), 264–266 (in English).—The oxidation is shown to be quant. in 1·5 to 2·5 *N* HCl medium and in the presence of ICl as a catalyst. A method of estimating Ce^{4+}, based on the reduction to Ce^{3+} by excess of KSCN and potentiometric titration of the excess against $KMnO_4$, is described. E. G. CUMMINS

1474. **The use of dioctyl phosphoric acid extraction in the isolation of carrier-free ^{90}Y, ^{140}La, ^{144}Ce, ^{143}Pr and ^{144}Pr.** D. F. Peppard, G. W. Mason and S. W. Moline (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *J. Inorg. Nuclear Chem.*, 1957, **5** (2), 141–146.—The values of *K* at 22° ± 2° are tabulated for the nuclides and ^{90}Sr and ^{140}Ba in systems comprising dioctyl phosphate in toluene or *n*-heptane and an aq. phase containing HCl or HNO_3. Procedures are given for the isolation of ^{90}Y from ^{90}Sr - ^{90}Y, ^{140}La from ^{140}Ba - ^{140}La, and Pr from Ce, and for the preparation of pure Ce. G. J. HUNTER

1475. **Vanadametry. Determination of thiocyanate. Use of iodine monochloride catalyst.** K. Bhaskara Rao, R. V. V. S. Murthi and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1957, **157** (3), 178–181 (in English).—The thiocyanate soln. is mixed with excess of standard sodium vanadate soln. and sufficient HCl (conc.) to give a soln. of the reagents in 1:1 HCl. The catalyst is added and the mixture kept at room temp. for 10 to 15 min. The excess of vanadate is back-titrated with standard $(NH_4)_2SO_4.FeSO_4$ soln., with N-phenylanthranilic acid as indicator. The reaction is quantitative and unaffected by atmospheric oxygen. L. S. ADLER

1476. **Determination of thiocyanate by potassium bromate.** M. K. Joshi (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **157** (3), 192–194 (in English).—The thiocyanate - bromate reaction has been re-investigated. Accurate and reproducible results are obtained when the overall HCl content at the end of the titration is 1·5 to 3·0 *N*. The end-point is determined either potentiometrically or with methyl orange indicator. L. S. ADLER

1477. **Use of heteropoly compounds for gravimetric and volumetric determination of germanium.** Z. F. Shakhova and R. K. Motorkina (Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (2), 183–193.—Improvements in the gravimetric method of determining Ge as pyridine molybdogermanate are given and a volumetric finish is described. A gravimetric method based on pptn. as guanidine molybdogermanate and a volumetric method based on quinoline molybdogermanate are also described, and it is shown that molybdovanadogermanic and tungstovanadogermanic acids cannot be used for the gravimetric and volumetric determination of Ge. *Use of the pyridine complex*—To the soln. (10 to 15 ml) of Ge neutralised to thymol blue indicator are added 2 ml of 10% H_2SO_4 and 10 ml of 5% ammonium molybdate soln. After 10 min. at 50°, 4 ml of conc. HCl and 5 ml of a 10% soln. of pyridine hydrochloride, prepared by diluting 10 ml of pyridine and 10 ml of conc. HCl to 100 ml, are added. After 3 to 4 hr. (or longer with small amounts of Ge) the ppt. is collected (the soln. above the ppt. should be clear) and washed four or five times with, in all, 50 to 60 ml of a soln. containing 12 g of NH_4NO_3, 10 ml of dil. HNO_3 (sp. gr. 1·2) and 0·5 g of pyridine nitrate (or its equivalent as chloride) in 250 ml of water. The ppt. is dried to const. wt. at 150° to 160°. The factor (empirical) for calculating to GeO_2 is 0·04968. For the volumetric finish the ppt. is collected in a fine ashless paper, and the washings are carefully observed for the first indication of a yellow colour instead of pink. The ppt. with the filter is placed in a flask and treated with 20 to 40 ml of 0·1 *N* NaOH and the excess is titrated with 0·1 *N* HCl in the presence of a mixture of cresol red and thymol blue. With amounts of GeO_2 between 0·5 and 11 mg the error is $\simeq \pm 0{\cdot}1$ mg. *Use of the guanidine complex*—To the soln. (10 to 30 ml) neutralised to thymol blue are added 2 ml of 5% ammonium molybdate soln. for each mg of GeO_2 and sufficient 10% H_2SO_4 to give a concn. $\leqslant$ 0·2 to 0·1 *N* (2 ml with a vol. of 20 ml). After 10 min., 2 ml of conc. HCl and 20 ml of 20% guanidine sulphate soln. (or 20 ml of saturated guanidine nitrate soln.) are added. The ppt. is collected in a filter crucible after 12 hr. or longer, and washed with 0·5% guanidine sulphate soln. or saturated guanidine molybdogermanate soln. The ppt. is dried at 130° to 140°. The factor (theoretical) for calculating to GeO_2 is 0·04897. *Use of the quinoline complex*—To the soln. (25 ml) containing 2 to 20 mg of GeO_2 neutralised to thymol blue are added 5 ml of 10% H_2SO_4, 4 to 10 ml of 5% ammonium molybdate soln. and water to give 50 ml. After 10 min., 4·5 to 5 ml of conc. HCl and, dropwise, 20 ml of 2% quinoline soln. are added. After 3 hr. the ppt. is collected and washed with a soln. prepared by adding 4 ml of 2% quinoline hydrochloride soln. and 4 or 5 drops of a mixture of cresol red and thymol blue soln. to 300 ml of water. When the washings show a yellow colour the ppt. is removed and dissolved in 0·2 *N* NaOH, the excess of which is then titrated with 0·1 *N* HCl. G. S. SMITH

1478. **Rapid semi-micro gravimetric method for lead.** E. Popper, N. Ariton and R. Popa-Crăciuneanu. *Stud. Cercet. Chim., Cluj*, 1956, **7** (1–4), 85–88.—Lead is pptd. as a salt by an alcoholic soln. of 1:2-di(phenylthiocarbamoyl)hydrazine. The method is exact, sensitive and convenient, and gives good results at concn. down to 200 μg per ml. H. SHER

1479. **Volumetric determination of lead by the method of internally complexed compounds.** M. E. Tsimbler and V. I. Derenovskiĭ. *Trudy Kievsk. Gidromelior. Inst.*, 1956, (5), 181–184; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,861.—A method is evolved for the determination of Pb as the tartrate complex formed according to the equation—

$$Na_2C_4H_4O_6 + Pb(NO_3)_2 + NaOH = NaPbC_4H_3O_6 + 2NaNO_3 + H_2O.$$

The reagent for obtaining the tartrate complex is a mixture of 0·1 to 0·05 *N* K Na tartrate and 0·085 to 0·042 *N* KOH in the ratio 1:0·85. One mol. of K Na tartrate reacts with one mol. of $Pb(NO_3)_2$. To the reagent (20 to 40 ml) add the Pb^{2+} soln., with stirring. The end-point of the titration is the appearance of a cloudiness. C. D. KOPKIN

1480. **Determination of small quantities of lead in minerals and rocks.** N. Iordanov and L. Kocheva. *Izv. Khim. Inst. Bulgar. Akad. Nauk*, 1956, **4**, 327–339; *Ref. Zhur., Khim.*, 1957, Abstr. No. 51,591.—In establishing the geological age of minerals and rocks, two methods of determining Pb are used. (*i*) Mix equal vol. of the sample and spectrally pure C and ignite in a quartz tube (length 6 cm, diam. 8 to 10 mm) at 950° to 1100° and 1 to 2 mm pressure. Dissolve the sublimate in 58% HNO_3 (0·65 ml), dilute to 20 ml, add 1 ml of a $CuSO_4$ soln. (30 mg of Cu) and electrolyse. Dissolve the pptd. PbO_2 in a mixture of two or three drops of 58% HNO_3 and one drop of H_2O_2 and evaporate the soln. to dryness. Dissolve the residue in a 10% $SrCl_2$ soln. in 0·1 *N* HCl, add one drop of a 0·5% gelatin soln. and determine Pb polarographically. With 1 to 10 μg of Pb work in a vol. of 0·1 to 0·4 ml (error 8 to 10%), and with >30 μg of Pb in a vol. of 1 to 4 ml (error 3 to 5%). (*ii*) Dissolve the sample in a mixture of H_2SO_4 and HF, add 1 ml of a soln. of $HgCl_2$ (50 mg), add water to 10 or 15 ml, acidify with 0·1 *N* HCl and saturate with H_2S. Filter off the ppt. of HgS and PbS, dissolve it in aqua regia, evaporate the soln. to dryness and heat the residue in a quartz crucible at 350° to 400° until all the S and HgS has sublimed. Determine Pb in the residue polarographically. The error in determining 50 μg of Pb is ≃ 5%. C. D. KOPKIN

1481. **Quantitative determination of lead in enamel paints containing a lead drier.** V. P. Kondratova and V. I. Petrashen'. *Nauch. Trudy Novocherkas. Politekhn. Inst.*, 1956, **27** (41), 211–212; *Ref. Zhur., Khim.*, 1957, Abstr. No. 51,593.—The method of determining Pb by electrolysis of a nitrate soln., previously recommended for the analysis of slags (Fainberg, "Technical analysis of the ores of noble metals," 1946, p. 307), is used in the determination of Pb in enamel paints containing 0·2 to 1% of Pb in organic combination. A rapid method is evolved for the preliminary treatment of the enamel paint for analysis (by heating samples of ≯2 g in a muffle-furnace at 500°), allowing a reduction of 7 or 8 hr. in the time taken to dissolve the paint. Satisfactory results are obtained.
C. D. KOPKIN

1482. **Simple method for the separation of titanium, niobium and tantalum from hard metals.** E. Lassner and H. Weisser (Metallwerke Plansee G.m.b.H., Reutte-Tirol, Austria). *Z. anal. Chem.*, 1957, **157** (5), 343–345.—An aq. NH_3 pptn. technique is described. Co-pptn. of small amounts of W, Fe and Co are prevented by the addition of EDTA and glycerol. This method is suitable for industrial application. E. G. CUMMINS

1483. **Determination of oxygen in titanium by the micro bromination method.** Mitsunao Takahashi, Makoto Kawane and Tetsuo Mitsui (Engng Res. Inst., Univ. Kyoto, Japan). *Mikrochim. Acta*, 1957, (5), 647–657 (in English).—The bromination method of Codell and Norwitz (*cf. Anal. Abstr.*, 1956, **3**, 94) for the determination of O in titanium has been modified. The sample (1 g) is mixed with graphite (0·8 g); A is used as carrier gas; the CO_2 is absorbed in soda - asbestos; last traces of bromine compounds are removed by a soda tube and silver gauze at 650°. The results agree well with those by the vacuum-fusion method for two samples of commercially pure titanium rod and plate.
A. R. ROGERS

1484. **Eriochrome black T and carminic acid as indicators in the complexometric determination of zirconium.** V. G. Goryushina and E. V. Romanova. *Zavod. Lab.*, 1957, **23** (7), 781–784.—Both carminic acid and Eriochrome black T (**I**) can be used as indicators in boiling acid (2 *N* HCl) soln. for the titration of Zr with EDTA (disodium salt) without interference from large amounts of Al, Bi, rare earths, Ti, U, etc., and from Fe (100 mg in 50 ml) and Cu (200 mg) if reduction with $SnCl_2$ is first carried out. To determine Zr in its salts, 0·5 g of sample is dissolved in 50 ml of dil. HCl (1 : 5) and then, with salts other than K_2ZrF_6, 10 ml is mixed with 20 to 30 ml of dil. HCl (1 : 5) and a mixture (1 : 50) of **I** and NaCl, and titrated while boiling with 0·05 *M* EDTA, the titre of which has been established against a standard soln. of Zr. When the bluish-violet colour changes to violet - red the soln. is again heated and the titrant is added dropwise. With K_2ZrF_6, 10 ml of the soln. is evaporated to dryness with 5 ml of dil. H_2SO_4 (1 : 1), the residue is dissolved in 20 to 30 ml of dil. HCl (1 : 5), and the titration is carried out as described above. To determine Zr in concentrates, the sample (0·2 g) is fused with 2 to 3 g of KHF_2, finally at 900° to give a transparent melt, the cooled melt is heated with 5 to 7 ml of dil. H_2SO_4 (1 : 1) and then more strongly until fuming ceases. It is then heated at 500° to 600° to give a transparent melt, the cooled melt is extracted with 150 to 200 ml of hot water and the soln. is heated with 10 to 15 ml of HCl until it is clear. Excess of aq. NH_3 is added, and the ppt. is filtered off, washed with hot 2% NH_4NO_3 soln., and dissolved in 100 ml of dil. HCl (1 : 5). An aliquot is taken for the titration. The end-point is stated to be very sharp, and amounts of Zr within the range of 1 to 100 mg can be titrated.
G. S. SMITH

1485. **The analysis of zirconium - cerium binary alloys.** G. W. C. Milner and G. W. Sneddon (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 1654, 1957, 12 pp.—The procedure described is suitable for alloys containing from 1 to 99% of either Zr or Ce. The sample soln. is prepared by dissolution of the alloy (1 to 3 g) in HCl (1 + 1), HF and H_2SO_4 (1 + 1), followed by evaporation of the soln. to fuming (to remove Cl^- and F^-) and dilution to 100 ml. Cerium is determined in an aliquot (≡ 10 to 70 mg of Ce) by oxidation with ammonium persulphate at 100°, followed by titration with 0·02 *N* $Fe(NH_4)_2(SO_4)_2$ at 20° (ferroin indicator). Zirconium is determined volumetrically in another aliquot (≡ 10 to 50 mg of Zr) by adding a small excess of 0·02 *N* EDTA (disodium salt), adjusting the pH of the soln. to 2·2 (to avoid interference by Ce) and then titrating the excess of EDTA with 0·02 *N* $FeCl_3$ to a photometric end-point, potassium benzohydroxamate (2·5% aq. soln.) being used as indicator. The precision is ≃ 1% for each element. Turnings, not drillings, should be taken from the alloy. W. J. BAKER

1486. **Determination of tin in zirconium and its alloys.** D. F. Wood and R. T. Clark (I.C.I. Ltd., Metals Div., Kynoch Works, Witton, England). *Analyst*, 1957, **82**, 624–630.—The sample is dissolved in a specified amount of conc. HCl and fluoroboric acid, the acid content is then adjusted to 1 + 1 with respect to HCl, 15 ml of a $TiCl_3$ soln. (1 ml ≡ 10 mg of Ti) is added, the liquid is boiled gently with aluminium turnings in a stoppered flask with a delivery tube leading into a soln. of $NaHCO_3$, and

then allowed to cool, the $NaHCO_3$ seal being maintained. The delivery tube is removed, NaI (2 g) is added and the re-stoppered flask is shaken. The liquid is then titrated with standard KIO_3 soln., starch being used as indicator. If Cr is present in excess of 0·5% in the range 0·5 to 2% of Sn or 0·25% in the range of 2 to 20% of Sn, an aliquot of the sample soln. is taken to bring the content of Cr within these limits. An alternative procedure is described for use in the presence of Cr, involving preliminary separation of Sn as sulphide, dissolving the ppt. in HCl and proceeding as before. The method is accurate over the range 0·01 to 20% of Sn. Interference by Fe and Ni (20%), W (10%) and Cu, Mo and V (5%) is negligible. The standard deviation is $\pm$ 0·015 at the level of $\simeq$2% of Sn.

A. O. JONES

1487. **Photometric determination of thorium with quercetin.** E. Tomic and H. Khalifa (II. Chem. Inst., Univ. Wien). *Mikrochim. Acta*, 1957, (5), 668–676 (in German).—Evaporate to dryness a sample that contains Th (2 to 60 μg) and $>$ a 10-fold excess of U or rare earths and $<$100 μg of Fe. Dissolve the residue in 0·1 *N* HCl (2 ml). Add 0·4 *M* ascorbic acid (1 ml), KSCN (trace), and 0·1% ethanolic quercetin soln. (3 ml), shake, and dilute to 10 ml with acetate buffer of pH 5·6. Determine the extinction in 1-cm cells at 425 to 450 mμ. For the determination of smaller amounts of Th (1 to 10 μg), use a 0·01% quercetin soln. and measure in 3·4-cm cells.

A. R. ROGERS

1488. **Determination of ammonia with cupric carbonate.** R. C. Blinn and F. A. Gunther (Univ. of Calif. Citrus Exp. Sta., Riverside, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1882–1883.—Two methods are described for application to ammonia-releasing formulations. The first, titrimetric, procedure is based on the dissolution of pptd. cupric carbonate by titration with a clear extract of the ammonium salts of the sample; the end-point is the formation of a clear blue soln. of the cupric ammonium salt. The concn. of ammonia is determined by means of standard curves. The second, colorimetric, method is based on absorptiometric measurement at 700 mμ of the blue cupric ammonium complex in alkaline soln., produced by mixing soln. of Na_2CO_3, $CuSO_4$, the ammonium salt sample and $Ca(NO_3)_2$. Results from synthetic mixtures showed recoveries within $\pm$2% of theoretical.

E. G. CUMMINS

1489. **Determination of small quantities of ammonia in gas mixtures.** Y. Kauko and S. Döger (Phys.-Chem. Inst., Univ. Ankara, Turkey). *Acta Chem. Scand.*, 1957, **11** (5), 804–810.—An accurate potentiometric method for the measurement of the partial pressure of ammonia, p_{NH_3} ($<10^{-3}$ atm.), in gas mixtures has been developed. The p_{NH_3} is evaluated from measurement of the pH of an NH_4Cl soln. in equilibrium with the unknown p_{NH_3}. Calibration is effected by reference to a gas mixture with known $p^{\circ}_{NH_3}$.

L. S. ADLER

1490. **Spectrochemical determination of trace impurities in commercial-grade ammonium chloride.** K. W. Beyer and O. T. Aepli (Pennsalt Chemicals Corp., Wyandotte, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1779–1780.—A 25-g sample of NH_4Cl is placed in a 100-ml evaporating dish and thoroughly wetted with H_3PO_4 (1%) (10 ml). After the sample has been dried at 130° for 1 hr., the cake is broken up into quarter-inch pieces, and sublimed to remove NH_4Cl. The residue is dissolved in 5 ml of redistilled HNO_3 and evaporated almost to dryness. Silicic acid is removed by further evaporation with 1 ml of HF and a few drops of redistilled HNO_3, and the final soln. is prepared by the addition of the minimum amount of HNO_3, 1·0 ml of buffer soln. (5·0 mg of Bi and 50 g of $NaNO_3$ per litre) and 1 ml of internal standard soln. (1·7 g of Zn acetate per litre). This soln. is placed on an impervious (kerosine-washed) electrode and dried for 1 hr. before being burnt in a 2300-V arc at 5 amp. Detailed determinations of the impurities Cu, Fe, Ni and Pb are tabulated.

E. G. CUMMINS

1491. **Ultra-violet spectrophotometric determination of nitrate. Application to analysis of alkaline-earth carbonates.** R. Bastian, R. Weberling and F. Palilla (Sylvania Electric Products Inc., Flushing, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1795–1797.—Measurements of the absorption at 203, 210 or 220 mμ of a dil. soln. of the carbonate mixtures in perchloric acid (5 ml of free perchloric acid per 100 ml) are used to determine the nitrate content. Seventeen non-interfering and twelve interfering ions are listed and the elimination of cation interference is discussed. Nitrate present at the 0·1 to 1·0% level in the triple carbonate of Ba, Sr and Ca has been determined by this method, and the tabulated results compare favourably with those obtained by using the brucine method after dissolving the sample in acetic acid and passing the soln. through a cation-exchange resin [Amberlite 1R-120(H)] to remove alkaline earths.

E. G. CUMMINS

1492. **Determination of arsenic in lead of high purity by means of a complexone.** Yu. Yu. Lur'e and A. N. Minenko (State Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1957, **23** (7), 785–786.—The method is suitable for determining $\simeq$0·0001% in lead on a sample wt. of 1 g. The sample is dissolved in 10 ml of dil. HNO_3 (1:3), oxides of N are boiled off, 1·5 ml of $Fe(NO_3)_3$ soln. (10 mg of Fe per ml), 40 ml of 7% EDTA (disodium salt) soln. and 45 ml of 7·5% $Ca(NO_3)_2$ soln. are added, and a small excess of aq. NH_3 is added to the boiling soln. to precipitate $Fe(OH)_3$ together with any As. The ppt. is collected and the As, after volatilisation as AsH_3, is determined colorimetrically.

G. S. SMITH

1493. **Determination of oxygen in niobium.** W. R. Hansen and M. W. Mallett (Battelle Memorial Inst., Columbus, Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1868–1869.—A diffusion-extraction method for removing and estimating the oxygen in niobium wires, by heating them to 2000° at 10^{-5} mm of Hg in a standard vacuum extraction apparatus, is described. The mean deviation of the results at the 0·019 wt. % oxygen level is $<$0·001 wt. %.

E. G. CUMMINS

1494. **Separation of niobium from tantalum, titanium, tin and antimony by means of 8-hydroxyquinoline.** G. K. Belekar and V. T. Athavale (Chem. Div., Atomic Energy Estab., Bombay). *Analyst*, 1957, **82**, 630–634.—The purified oxides ($>$0·2 g) (with or without oxides of Sn and Sb) are thoroughly fused with $NaHSO_4$, and the melt is extracted with ammonium tartrate soln. and aq. NH_3; any insol. matter is re-fused and re-extracted. The soln. is diluted, the concn. of ammonium tartrate is increased to 2%, NH_4Cl is added and the pH is adjusted to 6·0. The warmed liquid is treated with

oxine soln. and, after digestion of the mixture on the water bath for 30 min., the ppt. is collected and washed free from SO_4^{2-} with a cold NH_4Cl and tartrate soln., the pH of 6 being maintained throughout. The ppt. is then dried under a layer of solid oxalic acid, ignited and weighed. A re-pptn. is made by the same procedure and a final purification by heating with HF and H_2SO_4 and extraction of insol. impurities with conc. HCl. Tantalum and Ti can be recovered from the filtrate by pptn. with tannic acid, Sb and Sn, if present, being first removed as sulphides. Spectrographic examination showed that the niobium fraction was contaminated with Ta and *vice versa*, but the purity of each fraction was >97·5%. A. O. JONES

1495. **Direct titration of sulphate.** M. Q. Freeland and J. S. Fritz (Iowa State Coll., Ames). *U.S. Atomic Energy Comm.*, Rep. ISC-667, 1956, 32 pp.—A method is proposed in which sulphate is titrated directly with standard barium soln. The end-point is indicated by the adsorption of alizarin red S or thorin on the $BaSO_4$ with distinct colour change from yellow to pink. The sulphate sample must contain 30 to 40% methanol for these indicators. Co-pptn. errors are greater than for gravimetric sulphate methods, but most of these can be avoided by the preliminary removal of cations with an ion-exchange column. In the absence of interfering anions the ion-exchange titrimetric method is as precise as the gravimetric method and considerably faster. CHEM. ABSTR.

1496. **Determination of sulphate in aluminium oxide by amperometric titration.** S. N. Aleksandrov and S. A. Alekseev. *Khim. i Tekhnol. Topliva*, 1956, (12), 65–67; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,882.—Shake 5 g of Al_2O_3 vigorously with 1% NaOH soln. (20 ml) for 15 min., set aside for 1 or 2 min., filter off and wash the ppt. with 10 or 15 ml of water, transfer the filtrate and washings to an electrolyser, neutralise with HNO_3, dilute to 30 or 35 ml, add acetone (15 ml), stir and titrate with standard $Pb(NO_3)_2$ soln. (41·4 g per litre) at 1·4 V *vs.* the S.C.E. If, after adding 0·2 or 0·3 ml of the soln., no cloudiness of $PbSO_4$ is formed, the concn. of sulphate in the soln. is <0·1%, and the titration is not completed. If after the addition of the first drops of $Pb(NO_3)_2$ soln. there is a heavy cloudiness and after adding 5 or 6 ml there is no inflection point, the determination is repeated with a smaller sample (1 g) of Al_2O_3. The end-point is found by the usual method. The error is up to ± 5%. Sulphate from 0·1 to 10% in Al_2O_3 can be determined. C. D. KOPKIN

1497. **Spot test for sulphites based on the induced oxidation of cobalt - azide solutions.** P. Senise (Dept. de Quím., Univ. São Paulo, Brazil). *Mikrochim. Acta*, 1957, (5), 640–643 (in English).—Treat one drop of 1·5% $Co(NO_3)_2.6H_2O$ soln. at pH 5 on Whatman No. 1 paper with one drop of saturated sodium azide soln. and add one drop of the test soln. (pH 5 to 6) to the blue - violet spot. If sulphite is present, a pale-yellow colour appears which changes to blue on the addition of a 0·2% soln. of *o*-tolidine in 2% acetic acid. The limit of identification is 0·5 μg of $NaHSO_3$ in 0·05 ml of soln. Remove S^{2-} by pptn. with Cd^{2+} soln. In the presence of I^-, HPO_3^{2-}, PO_3^{3-} or MoO_4^{2-}, liberate SO_2 by addition of dil. HCl to the sample and test the evolved gas. In the presence of large amounts of $S_2O_3^{2-}$, add $HgCl_2$ soln. to the sample and after 1 min. acidify and test the evolved SO_2. A. R. ROGERS

1498. **Assay of sodium hydrosulphite [dithionite].** Committee on Analytical Methods, Amer. Ass. Text. Chem. Colorists. *Amer. Dyest. Rep.*, 1957, **46** (12), P443–447.—Various methods were reviewed and a detailed study was made of the ammoniacal $CuSO_4$, the iodine - formaldehyde, and the Rubine R methods. Each is described fully. The three methods all give essentially comparable results, but the second is recommended because of its simplicity. It is applicable in all cases except when there is a significant amount of thiosulphate, sulphite or other oxidisable impurity not inactivated by formaldehyde. S.C.I. ABSTR.

1499. **Spectrophotometric determination of traces of selenium.** Yukio Suzuki, Keitaro Nishiyama, Yutaka Matsuka, Shigeru Kuwai, Masahiro Oe, Hideo Hujiwaka, Tsuyoshi Wakatsuki, Takeshi Nakanishi and Masahiro Doi (Sch. of Med., Tokushima Univ.). *Shikoku Acta Med.*, 1957, **11** (1), 77–79.—A simple and precise method for the spectrophotometric determination of traces of selenium in biological materials, soil, air and water is described depending on the colour produced with 3;3′-diaminobenzidine. The reagent can be used for the detection of selenium in paper chromatography. N. E.

1500. **Eddy current electrical conductivity measurement as an analysis for molybdenum in binary molybdenum - uranium alloys.** R. E. Cofield (Union Carbide Nucl. Co., Y-12 Plant, Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-1152, 1957, 32 pp.—The principles involved in a non-contact, electromagnetic induction or eddy current method of measuring the electrical conductivity of a solid are discussed and a simple eddy current conductivity meter is described. The problems involved in the conductivity analysis of binary alloys, including the general relationships between electrical conductivity and alloy structure, composition and metallurgical conditions, are discussed. The method has been used for the determination of Mo in U - 2% Mo alloys with agreement with chemical analysis within ±0·128% of Mo. Such a method has many potential uses in analytical chemistry and metallurgy. NUCL. SCI. ABSTR.

1501. **New type of analysis of small quantities of tervalent chromium based on the extinction of the luminescence of uranium.** I. E. Starik, E. F. Starik and G. V. Kostȳrev. *Trudȳ Radiev. Inst. Akad. Nauk, SSSR*, 1956, **7**, 111–113; *Ref. Zhur., Khim.*, 1957, Abstr. No. 48,293.—The method is based on the estimation of the decrease in the uranium luminescence in a bead obtained by fusion with NaF, in the presence of Cr^{3+}. Dip the bead in a standard soln. of $UO_2(NO_3)_2$ ($2·5 \times 10^{-6}$ g per ml), fuse, and measure the luminescence. Then dip the bead in the soln. of Cr^{3+} to be analysed, fuse, and again measure the luminescence. With $>10^{-5}$ g of Cr per ml there is a straight-line proportionality between the concn. of U (%) and the log of the concn. of Cr in the bead. With 10^{-8} to 7×10^{-6} g of Cr, the error of the determination is <16%. With $<10^{-6}$ g of Cr, add to 5 or 10 ml of the soln. $n \times 10^{-7}$ g of $UO_2(NO_3)_2$ and a soln. of 25 mg of NaF, evaporate the soln. to dryness, fuse the residues to obtain beads, the luminescence of which is then measured; from the calibration curve the concn. of Cr is found ($\simeq n \times 10^{-6}$ g per ml). C. D. KOPKIN

1502. **Luminescence method of determining uranium without removal of extinguishing substances.** L. Ya. Atrashenok, L. L. Voloshenko and A. Ya. Krÿlov. *Trudÿ Radiev. Inst. Akad. Nauk, SSSR*, 1956, **7**, 126–136; *Ref. Zhur., Khim.*, 1957, Abstr. No. 48,294.—The suppression of the uranium luminescence in NaF by certain elements is studied, and it is established that there is additivity of extinguishing power for elements possessing this property of one type (the systems Fe - Ca, Al - Ca and Fe - Al). The silicates of Fe, Al and Ca show a greater extinguishing power than would be expected from the additivity of that of the elements forming them; this is explained by the screening action of the solids formed in the presence of Si. It is established that the suppression of the uranium luminescence due to various elements depends on the ratio of their concn. With $> 10^{-5}$ g of U per g, its determination by the luminescence method, by fusion with NaF, is possible without removal of extinguishing substances. The method has been tested on leucocratic granites, limestone and clay; in the analysis of rich ores it is recommended that they be dissolved and U determined in the soln. by dipping the bead therein. Direct fusion with NaF is used with a uranium content in ores of 0·001 to 0·1%, and in associated minerals with a uranium content of >0·001%. C. D. KOPKIN

1503. **Absorption spectra of the complexes of uranium(VI) with some β-diketones.** H. I. Feinstein (U.S. Geological Survey, Washington, D.C., U.S.A.). *Microchem. J.*, 1957, **1** (2), 237–244.—The absorption spectra of the coloured complexes given with U^{VI} with four diketones have been studied for their use in the colorimetric determination of U; optimum conditions are given. The uranium soln. (5 ml, up to 10 p.p.m.), or a blank of 7% HNO_3, is treated with 26 ml of 95% ethanol, 15 ml of water and 1 ml of 1% diketone soln. in 95% ethanol. The pH is adjusted with aq. NH_3 (1:1 or 1:10) or 7% HNO_3, the vol. is made up to 50 ml and the absorbance read at once. Beer's law was obeyed by all the complexes at optimum pH and wavelength. The optimum pH, wavelength and ϵ for the four reagents are—2-thenoyltrifluoroacetone, 5·5, 380 mμ, 20,600; 2-thenoylperfluorobutyrylmethane, 6·5, 385 mμ, 21,400; 2-furoyltrifluoroacetone, 4·5, 330 mμ, 31,200; 2-furoylperfluorobutyrylmethane, 6·0, 385 mμ, 24,000. For the thiocyanate and dibenzoylmethane complexes, values for ϵ were 4,000 and 19,000, respectively. Interference was not studied, but it is known that many elements interfere with dibenzoylmethane methods, and must be removed before determining U. R. E. ESSERY

1504. **Spectrographic analysis by the evaporation method. III. The role of the "carrier" in the spectrographic analysis of uranium.** A. N. Zaĭdel', N. I. Kaliteevskiĭ, G. G. Kund and Z. G. Fratkin. *Optika i Spektroskopiya*, 1957, **2** (1), 28–33; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,664.—In the determination of impurities in uranium oxides, the introduction of a "carrier," Ga_2O_3, which is stated to facilitate the evaporation of the impurities and at the same time increase the sensitivity of the analysis (Scribner and Mullin, *J. Res. Nat. Bur. Stand.*, 1946, **37**, 369) is criticised. The presence of Ga increases the sensitivity of the determination of certain elements, not because it facilitates the evaporation, but as a result of the influence on the plasma of the arc discharge. With the spark discharge the presence of Ga lowers the sensitivity of the analysis. Taking into account the fact that the analysis is made more complicated by the addition of the "carrier," and the danger of introducing impurities, the addition of Ga_2O_3 to standards and samples is not recommended. C. D. KOPKIN

1505. **Quantitative spectrographic analysis of the isotopic composition of enriched uranium.** A. V. Striganov, F. F. Gavrilov and S. P. Efremov. *Atomnaya Energiya*, 1957, (4), 337–344; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,702.—The spectra are excited in an a.c. arc at 5 amp. with arc gap 2·5 mm. The samples are analysed as aq. soln. of $UO_2(NO_3)_2$. The standards are prepared from salts of natural and enriched U which have been analysed mass spectrometrically for ^{234}U, ^{235}U and ^{238}U. Evaporate the soln. in glass dishes, add 0·06 ml of water for each mg of metallic U and add 0·001 ml of 2 *N* HNO_3 for each ml of soln. Transfer a drop of the sample soln. to the flat polished surface of a graphite electrode of 5-mm diameter, dry, and place in an oven at 150° to 200° for 10 min. The analysis is carried out by the uranium line at 4244·374 A, which has the isotopic shift 0·246 A. The calibration curves are constructed with the co-ordinates $\log I_5/I_8$ and $\log C_5/C_8$, where I_5 and I_8 are the intensities of the isotopic lines of ^{235}U and ^{238}U, and C_5 and C_8 are the concn. of the isotopes, relative to their total. For calculating the mutual superimposition of the lines and the background, measurement is made of the blackening to the right and left of the lines due to ^{235}U and ^{238}U, and also in the minimum between them. The curves intersect at the point corresponding to the concn. $C_5 = C_8 = 50\%$. The mutual superimposition bends the curve towards the concn. axis below 10% and above 90%. The graph is constructed from the fixed point $\log I_5/I_8 = 0$, $\log C_5/C_8 = 0$ and from one of the standards. For low and high concn. (3 to 10% and 90 to 97%) of ^{235}U it is advisable to use two standards. In this case to obtain straight lines it is necessary to allow for the background and the mutual superimposition. For low concn. of ^{235}U (0·7 to 3%) the curve is not linear, even when allowing for the background and the mutual superimposition of both lines. The coeff. of variation of a single determination for concn. of 4 to 90% is from ±2·2% to ±0·3%. The presence of the ^{234}U component or of other impurities does not introduce errors into the analysis. C. D. KOPKIN

1506. **Determination of uranium-235 by gamma scintillation spectrometry.** G. H. Morrison and J. F. Cosgrove (Sylvania Electric Products, Inc., Flushing, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1770–1771.—The method is based on the use of gamma scintillation spectrometry to measure the gamma photons emitted during natural radioactive decay. The limit of sensitivity of the method, which is rapid and non-destructive, is approx. 0·05% of ^{235}U. Accuracy and precision are within 1% over the concn. range 0·72% to 100% of ^{235}U. K. A. PROCTOR

1507. **Precision of the pyrohydrolytic determination of fluoride and uranium in uranyl fluoride and uranium tetrafluoride.** J. O. Hibbits (Union Carbide Nuclear Corp., Oak Ridge, Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1760–1762.—In the method described, superheated steam between 400° and 1000° is passed over the metal halide in a platinum or nickel reaction tube. The volatile acids are condensed and titrated against 0·1 *N*

NaOH, with phenolphthalein as indicator (0·5% in 50% aq. ethanol). The results are discussed statistically. E. G. CUMMINS

1508. **A new method of determining uranium, radium, thorium and actinium in fine particles of radioactive minerals.** G. Becquerel. *J. Phys. Radium*, 1956, Suppl. to **17**, 137A–142A.—This method permits the determination of U, Ra, Th and Ac in a sample of only 0·01 cu. mm, and emitting only one α-particle per sec. The sample was deposited in a thin layer on a nickel plate. A fluorescent screen (disperse ZnS activated by Ag in polystyrene and spread on a cellulose triacetate film) was placed at various distances, *d*, from the sample and the fluorescence was measured with a photomultiplier. The counting yield $T = [(2\cos^{-1}d/P)/\pi]\ [(1 - e^{-\lambda R})/(1 - e^{-\lambda P})]$, where P is the total range of the α-particle, R is remaining range after hitting the screen, and $1/\lambda$ is the mean distance between two centres excited by the same α-particle. The theoretical values of T were tabulated for U I, U II, Io, Ra, Th and Ac in equilibrium mixture with their decay products. The determination of Ra and Th was made by volatilising the decay products in a furnace at 800°, and following the growth of active deposit by using a screen which absorbed all the α-particles from the parent substance. Uranium and Ac were determined by measuring the energy of the α-particles. CHEM. ABSTR.

1509. **Analytical chemistry of plutonium.** C. F. Metz (Univ. of Calif., Los Alamos, N.M., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1748–1756.—The separation of Pu from its impurities by pptn., ion exchange and solvent extraction, and the determination of Pu by potentiometric titration, and spectrophotometric and radiochemical methods, are reviewed. Data on slightly sol. plutonium compounds, useful organic solvents, transport numbers, oxidation-reduction potentials, and polarographic half-wave potentials are tabulated. E. G. CUMMINS

1510. **Separation and determination of plutonium by liquid-liquid extraction.** F. L. Moore and J. E. Hudgens, jun. (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1767–1770.—The Pu^{IV} in soln., obtained by chemical treatment of neutron-irradiated uranium, can be extracted to the extent of 99 ± 3% from HNO_3 (0·5 to 1·5 *M*) by a 0·5 *M* soln. of 2-thenoyltrifluoroacetone in xylene. Any high γ-radiation can be washed out of the organic phase by aq. HNO_3 (10 *M*) and the Pu checked by an α-counter. Details of pre-treatment and theoretical work are given. E. G. CUMMINS

1511. **The lanthanum fluoride co-precipitation method for the determination of plutonium. The efficiency of precipitation of the lanthanum carrier as fluoride.** R. G. Monk (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2382, 1957, 12 pp.—The procedure recommended by Spence *et al.* (A.E.R.E. Report CRC-343) for the determination of trace amounts of Pu by co-pptn. of PuF_3 and LaF_3 ensures that >99·9% of the La is pptd. as carrier. It can be modified, without loss of this accuracy, by reducing the 17-hr. standing-period to 1 or 2 hr. and increasing the time of centrifuging to 5 min. The new procedure after the addition of 48% HF is—stir the soln. and centrifuge it for 2 min., add 0·15 ml of lanthanum nitrate soln. (1·6 mg of La per ml) dropwise and stir the supernatant liquor only. Allow the tube to stand for 1·5 hr. and then centrifuge it for 5 min. Remove all the supernatant liquor except 0·1 ml and stir the ppt. with 1 ml of 0·5 *M* HF. Centrifuge for 5 min. and again remove all the supernatant liquor except 0·1 ml. Transfer the ppt. to the counting tray, evaporate the slurry to dryness (i.r. lamp), and then determine the Pu by alpha-counting. The main errors in the determination are unrelated to the incomplete pptn. of LaF_3. W. J. BAKER

1512. **Detection of fluoride ion.** H. Ballczo and H. Weisz (II. Chem. Inst., Univ. Vienna). *Mikrochim. Acta*, 1957, (5), 751–754 (in German).—Chromazurol S (**I**) is liberated from the Al-**I** complex by the action of F^- at pH 4·3 (*cf.* MacNulty and Woollard, *Anal. Abstr.*, 1956, **4**, 3041). The ring-oven technique is used to wash out the liberated **I** with ethanol on filter-paper and re-convert it into the blue aluminium lake. The method will detect 0·005 μg of F^-. A. R. ROGERS

1513. **Micro-determination of fluorine.** J. Samachson, N. Slovik and A. E. Sobel (Jewish Hosp. of Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1888–1891.—Fluoride is distilled from H_2SO_4 in the apparatus illustrated, which is an adaptation of that of Huckabay *et al.* (*cf. Anal. Chem.*, 1947, **19**, 154), and determined colorimetrically by using a stable modified alizarin reagent; the extinction is measured at 525 mμ. Blanks of <5 μg were obtained. Since chloride interference is excluded by addition of Ag_2O, the analysis of bones or blood is possible. E. G. CUMMINS

1514. **Potentiometric determination of iodide and iodine in iodine-iodide solutions.** J. Majer and E. Tomásch (Inst. Pharm. Chem., Komenský Univ., Bratislava, Czechoslovakia). *Českosl. Farm.*, 1957, **6** (7), 380–383.—Iodide is first determined by titration with $KMnO_4$, KIO_3 or $AgNO_3$ soln. Potassium cyanide, HCl or acetone is then added, and reacts with the iodine present to form an iodo deriv. and HI, which is titrated with the volumetric reagent used for determining iodide. Sulphuric acid (2 *N*) is used as the titration medium. Argentimetric titration can be used even in the presence of organic compounds. The mechanism of the oxidation is discussed. J. ZÝKA

1515. **Determination of small amounts of iodide in the presence of chloride by potentiometric titration.** R. H. Stokes and L. A. Woolf (Univ. of New England, Armidale, N.S.W., Australia). *Anal. Chem.*, 1957, **29** (12), 1883–1885.—The detrimental effect of small amounts of Cl^- (<0·1 *M*) on the iodide end-point is explained. Because of the existence of an argentochloride complex, the addition of excess of Cl^- enables the iodide to be accurately determined in these circumstances. E. G. CUMMINS

1516. **Polarographic determination of manganese in copper alloys by oxidation on a platinum anode.** E. M. Skobets and N. I. Belinskaya (Ukr. Agric. Acad.). *Zavod. Lab.*, 1957, **23** (7), 791–793.—The sample (0·3 g) of copper alloy is dissolved in 2 to 3 ml of dil. HNO_3 (1:1), the soln. is boiled to remove oxides of N, then diluted to 50 ml, and 5 ml is transferred to a polarographic cell (with a platinum-wire anode and an agar connection to a S.C.E. as cathode) already containing 20 ml of *N* NH_4Cl in 0·17 to 0·25 *N* aq. NH_3. The polarogram is taken to include the peak of the wave due to Mn (the

$E_{\frac{1}{2}}$ varies with concn. and other factors), the platinum electrode is washed with hot dil. H_2SO_4 (1:1) and then with water, an accurately measured amount of 0·1 *M* $MnSO_4$ is added to the soln. and the polarogram is again taken. The wave height is proportional to the amount of Mn present.

G. S. SMITH

1517. **Iodimetric determination of potassium permanganate by arsenous oxide following iodide reduction in presence of borax - boric acid buffer and barium chloride.** M. G. Bapat and B. Sharma (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **157** (4), 261–263 (in English).—The permanganate is reduced by KI to MnO_2, which is coagulated by the addition of $BaCl_2$; this prevents any further reduction. The titration is completed with arsenous oxide, the amperometric dead-stop end-point technique being used. E. G. CUMMINS

1518. **Thiocyanate spectrophotometric determination of technetium.** C. E. Crouthamel (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1756–1760.—When reduced in an acid medium with thiocyanate, Tc^{VII} forms a red complex, which can be determined by absorptiometric measurement at 513 mμ; at this wavelength the mol. extinction coeff. is 52,200 ± 500. A yellow complex is also formed under these conditions, but its mol. extinction coeff. is approx. half that of the red complex. E. G. CUMMINS

1519. **Polarographic determination of ferric ions by ascorbic acid.** L. Erdey and A. Karsay (Inst. für Allg. Chem., Tech. Univ., Budapest, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 43–48.—Polarography may be used to detect the equivalence point in the titration of Fe^{3+}, in 0·1 *N* HCl, with 0·01 *N* ascorbic acid. The diffusion current is measured at 0·0 V *vs.* the S.C.E. Concn. of Fe^{3+} as low as 0·001 *M* can be determined. The error on 1 to 2 mg of Fe is < 1%. Oxidising agents, Cu and Ag interfere. G. H. FOXLEY

1520. **Spectrophotometric determination of iron in clay and limestone.** P. F. Lott and K. L. Cheng (Univ. of Missouri, Rolla, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1777–1778.—EDTA (disodium salt) (0·1 *M*, 10 ml) is used to complex the metal cations in an aliquot (5 ml) of the soln. of a clay sample (1 g, fused and digested with Na_2CO_3, filtered to remove silica, and diluted to 100 ml). On the addition of 3 ml of aq. NH_3 (14 *M*) and 4 ml of 30% H_2O_2, and dilution to 100 ml, the EDTA acts as a colour-forming agent with the Fe, and absorptiometric measurement is made at 520 mμ. Chromium and Co would interfere but their presence is revealed by colour reactions and a preliminary removal or masking is then carried out.

E. G. CUMMINS

1521. **The alcoholic-iodine method for the extraction of inclusions from steel.** J. E. Garside, T. E. Rooney and J. J. J. Belli (B.I.S.R.A., 11, Park Lane, London). *J. Iron St. Inst.*, 1957, **185** (1), 95–103.—The procedure described incorporates the many modifications of the original method (*cf.* Willems, *Arch. Eisenhüttenw.*, 1927–8, **1**, 655). The sample (10 g) is treated with 600 ml of a 10% soln. of iodine in methanol, in a nitrogen atmosphere, warmed to 65°, and stirred continuously until all the metal has dissolved (≃4 hr.). The residue is filtered off in a nitrogen atmosphere, ignited with the filter-paper at 1000° in a platinum crucible and analysed by standard semi-micro methods. As this procedure causes decomposition of both carbides and sulphides, leaving oxides unaffected, total-oxygen figures calculated from these determinations agree well with those from vacuum-fusion analyses. A similar operation may be carried out at 30°, and the residue, containing most of the non-metallic inclusions, used for X-ray analysis. J. O. LAY

1522. **Influence of particle size of separated silicates on the photometric determination of silicon [in iron].** W. A. Fischer and M. Wahlster (Max-Planck-Inst. f. Eisenforsch., Düsseldorf, Germany). *Arch. Eisenhüttenw.*, 1957, **28** (7), 379–381.—While the gravimetric method for the determination of Si in steel gives values for the total Si, the photometric method gives equivalent values only if the size of the silicate inclusions is < 6 μ. In the presence of larger aggregates (10 to 15 μ) formed in the cooling process the latter method is unsatisfactory.

E. STERN

1523. **A colorimetric method for the analysis of [determination of magnesium in] iron alloys.** A. M. Dymov and A. N. Shchelkunova. Report of a Conference on "The Production of Steel," publ. by Metallurgizdat, 1956, 417–432; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,849.—A colorimetric method is evolved for the determination of Mg in cast iron as the hydroxyquinolinate; Mn is separated from Mg as MnO_2 or pptd. as hydroxyquinolinate in acid soln. C. D. KOPKIN

1524. **Spectrochemical determination of lead in steel.** L. C. Flickinger, E. W. Polley and F. A. Galletta (The Youngstown Sheet and Tube Co., Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1778–1779.—Twyman's spectrographic technique for the analysis of liquid specimens (*cf.* "Metal Spectroscopy," Griffin, London, 1951, 240–241) has been adapted to measure the Pb (0·05 to 50·0%) in a milling of steel dissolved in HCl (1:1). An aliquot of the soln. is placed in a cupped graphite rod which is used as the lower electrode. An over-damped, multi-source discharge is used to provide excitation between this and an upper cone-tipped graphite 120° electrode. Calibration is based on synthetic standards working with the Pb 4057·82-A line and the Fe 4017·15-A line. The precision of the method is discussed. E. G. CUMMINS

1525. **The determination of antimony in cast iron.** R. C. Rooney (B.C.I.R.A., Alvechurch, Birmingham, England). *Analyst*, 1957, **82**, 619–623.—The sample is dissolved in dil. HNO_3 under controlled conditions, graphite and SiO_2 are separated by filtration and the Sb is determined separately in the residue and filtrate. The residue is ignited, evaporated with HF to remove graphite and SiO_2, and then fused with $KHSO_4$, and the melt is leached with water and heated to fuming-point with H_2SO_4. The digest is dissolved in water, treated with a KI - NaH_2PO_2 reagent and the free iodine (if any) is removed with $Na_2S_2O_3$. After adjustment of the vol., the extinction of the coloured iodide soln. is measured at 425 mμ and referred to a calibration graph. The original filtrate is boiled with $KMnO_4$, the pptd. MnO_2 is re-dissolved by addition of H_2O_2, the excess of which is removed by boiling, $KMnO_4$ is added to the boiling liquid, and the collected ppt. (with co-pptd. Sb) is washed with NH_4NO_3 soln., dissolved in HNO_3 and evaporated to fuming-point with H_2SO_4. After dilution to a specified vol. the iron content is determined by a micro spot-test (described), and if this is > 0·05 g the MnO_2 pptn. is repeated. The soln. is diluted

and the determination is then as for the residue. Interference by Bi is negligible. A. O. JONES

1526. **Determination of uranium dioxide in stainless steel. X-ray fluorescent spectrographic solution technique.** L. Silverman, W. W. Houk and L. A. Moudy (N. American Aviation Inc., Canoga Park, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1762–1764.—The apparatus comprised an X-ray spectrograph with an X-ray tube (tungsten target) and a lithium fluoride analysing crystal; a scintillation counter and scaler were used to detect and record the counts. The sample was dissolved in aqua regia, then evaporated in the presence of HF and $HClO_4$ before adding $Sr(NO_3)_2$ as an internal standard and making up to known vol. The time taken to record 204,800 counts was measured at both the K_α line of Sr and the L_α line of U and the assay figures were obtained from the working curve, prepared from synthetic mixtures of UO_2 and stainless steel. The introduction of Sr into these mixtures, which contain 15 to 25% of UO_2, improved the accuracy of the method from 95% to 99·5%. E. G. CUMMINS

1527. **Micro-analysis with the aid of ion-exchange resins. XII. The detection of millimicrogram amounts of nickel with dithio-oxamide.** Masatoshi Fujimoto (Chem. Inst. Sci. Fac., Univ. Tokyo, Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (3), 274–278.—Conditions for the detection of Ni on the surface of an ion-exchange resin with dithio-oxamide have been investigated. Maximum sensitivity is attained if one granule of a strongly acid, moderately cross-linked, colourless cation-exchange resin (Dowex 50-WX8) is placed on a plate, moistened with one drop of the sample (diluted to a total salt concn. of <1 *N*) and one drop of *N* aq. NH_3, and set aside for 5 min. One drop of 0·1% dithio-oxamide in ethanol is then added, and the violet colour on the surface of the resin is observed with a lens after 2 to 3 min. Interference by Cr is prevented by preliminary oxidation to chromate, and interference due to Fe^{3+}, Co^{2+} and Cu^{2+} is avoided by passing the sample through a micro column of the chloride form of Dowex 1-X10 (100 to 200 mesh) with 12 *N* HCl, concentrating the eluate by evaporation to dryness and re-dissolving in water before the test. The sensitivity is 0·0021 μg of Ni at a limiting concn. of 1 in $1·9 \times 10^7$.

XIII. Detection of millimicrogram amounts of cobalt with nitroso-R salt. Masatoshi Fujimoto. *Ibid.*, 1957, **30** (3), 278–283.—One granule of a strongly basic, feebly cross-linked, colourless anion-exchange resin (Dowex 1-X1) is placed on a white plate, and treated with one drop each of 0·05% nitroso-R salt soln., 0·3 *N* Na acetate and the sample. After 5 min. one drop of 2 *N* HNO_3 is added and the plate is heated for 3 min. on a water bath, then allowed to cool. The resin acquires a red colour in the presence of Co. The HNO_3 treatment prevents interference by Ni and Cu, so that only Fe^{3+} significantly reduce the sensitivity, which is 0·0029 μg of Co at a limiting concn. of 1 in $1·4 \times 10^7$.

XIV. Detection of millimicrogram amounts of iron with 2:2′-dipyridyl. Masatoshi Fujimoto. *Ibid.*, 1957, **30** (3), 283–287.—One granule of a strongly acid, feebly cross-linked, colourless cation-exchange resin (Duolite C-60) is placed on a white plate and treated with one drop each of the sample, 3% hydroxylamine hydrochloride soln., *N* Na acetate and 0·5% ethanolic 2:2′-dipyridyl soln. The plate is covered with a watch-glass to keep out dust, and, after a few minutes, the red colour of the resin is observed through a lens. There is slight interference from Cu^{2+} and Co^{2+}. The sensitivity is 0·0025 μg of Fe at a limiting concn. of 1 in $1·6 \times 10^7$. A. B. DENSHAM

1528. **Photo-electric colorimetric determination of nickel in ferro-alloys.** Takuya Imai and Shinko Nagumo (Res. Lab., Nippon Denko Co. Ltd., Shinagawa-ku, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1957, **60** (5), 544–546.—The photometric determination of Ni (<500 μg per 100 ml) with dimethylglyoxime (**I**) was examined, with aq. Br soln. as an oxidising agent. Manganese is removed with $KClO_3$ in HNO_3. The sample is dissolved in HNO_3 ($\simeq 50$ ml) and treated with $KClO_3$ ($\simeq 4$ g), filtered, and diluted, and a portion treated with 50% citric acid (15 ml), aq. Br soln. (5 ml), aq. NH_3 soln. (5 ml) and **I** (1% in NaOH). The extinction is read at 540 mμ. Ferrosilicon and silicomanganese (0·5 g) are decomposed with HNO_3 (10 ml), HF (10 ml) and H_2SO_4 (1 + 1, 10 ml). The Ni - **I** complex is extractable with $CHCl_3$ or benzene from ammoniacal soln. in the presence of hydroxylamine hydrochloride. K. SAITO

1529. **Electro-migration on paper in the separation of ions. VI. [Ruthenium.]** Hari Gopal Mukerjee (Scottish Church Coll., Calcutta, India). *Z. anal. Chem.*, 1957, **157** (4), 268–272 (in English).—Ruthenium can be separated from Pt and Pa in a KCN, $NaNO_2$ or HCl medium. Its separation from Hg, Cu, Cd, Bi, Fe, Zn, Co and Ni in various electrolytes has also been effected. Separation sequences and the distances traversed by the cations of binary mixtures are tabulated. E. G. CUMMINS

1530. **Gravimetric determination of palladium with *cyclo*hexane-1:2-dione dioxime (nioxime).** N. K. Pshenitsyn and O. M. Ivonina. *Zhur. Neorg. Khim.*, 1957, **2** (1), 121–127; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,710.—Dilute the soln. of $PdCl_2$, containing 10 to 25 mg of Pd, to about 200 ml and add conc. HCl to 3% by vol. (pH 0·5). Warm on a water bath, add a 30% excess of 0·8% nioxime soln. and continue heating to coagulate the ppt. Filter the hot soln. through a weighed sintered-glass crucible. Wash the ppt. on the filter with hot water, dry it for 1 hr. at 110° to 120°, cool and weigh. It is possible to determine Pd in the presence of other platinum metals, in the ratio Pd:Pt:Rh:Ir of 10:10:2:1. No interference is caused by Ni, Cu, Fe^{3+} or Pb. The influence of a 10-fold quantity of Fe can be removed by the addition of a 5% EDTA (disodium salt) soln. C. D. KOPKIN

1531. **Separation of platinum, palladium, rhodium and iridium by paper electrochromatography.** W. M. MacNevin and M. L. Dunton (Ohio State Univ., Columbus, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1806–1809.—By using EDTA to complex Pd and Ir in alkaline soln. all four metals can be separated in limited amounts by horizontal paper electrochromatography. Certain combinations of the metals can be separated by continuous processes. K. A. PROCTOR

1532. **Separation and determination of the platinum metals on a micro scale.** A. D. Westland and F. E. Beamish (Univ. Toronto, Canada). *Mikrochim. Acta*, 1957, (5), 625–639 (in English).—Methods are described for the analysis of small crystals of the minerals of the Pt group, particularly iridosmines, and the "insoluble" portion of assay

beads and buttons. Separate Os by distillation from a H_2O_2 - H_2SO_4 soln. at 115° and determine Os in the distillate colorimetrically with thiourea (*cf.* Westland and Beamish, *Anal. Chem.*, 1954, **26**, 739) or gravimetrically with 2-phenylbenzothiazole (*cf.* Hoffman *et al.*, *Anal. Chem.*, 1953, **25**, 1091). Boil the soln. to decompose H_2O_2 and distil with $HBrO_3$ soln.; determine Ru in the distillate colorimetrically with thiourea. Concentrate the residual soln., boil for 30 min. with conc. HCl, dilute, add powdered tellurium and boil for 10 min. Reduce dissolved Te with $Na_2S_2O_5$, with HI as catalyst, filter through paper and retain the filtrate for the determination of Rh and Ir. Char the filter-paper and ppt., heat in a stream of H, ignite strongly in air, dissolve the residue in hot aqua regia, evaporate to dryness first with NaCl then three times with HCl, dissolve the residue in H_2O, and determine Pt and Pd colorimetrically with dimethyl-*p*-nitrosoaniline. Evaporate the filtrate to eliminate most of the SO_2. Separate Rh by pptn. with Sb from boiling H_2SO_4 soln. and determine colorimetrically with 2-mercapto-4:5-dimethylthiazole (*cf. Anal. Abstr.*, 1956, **3**, 3652). Remove dissolved Sb by distillation of $SbCl_3$ and determine Ir colorimetrically with $SnBr_2$ (*cf. Anal. Abstr.*, 1957, **4**, 1234).

A. R. ROGERS

1533. **Spectrographic analysis of welding fluxes.** E. S. Kudelya. *Avtomat. Svarka*, 1957, (1), 73–76; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,665.—In order partially to reduce the influence of possible inconsistencies in the total composition of samples of welding fluxes and standards, the samples are briquetted with powdered carbon or copper. The finely ground sample (0·5 g) is mixed with 1·5 g of powdered copper and pressed under a pressure of 2000 kg per sq. cm into a tablet of 100-mm diameter. The spectrum is excited at 2 amp. in an a.c. arc with a fixed copper electrode. The preliminary ignition is for 15 sec., arc gap 2 mm, and exposure 15 sec. The slit is illuminated without a condenser, the distance between the arc and slit being 100 mm. The spectra are photographed on a medium spectrograph with a slit width of 0·018 mm. Chemically analysed fluxes are used as standards. The analytical pairs of lines used (in A) and the limits of concn. (%, in brackets) are—Al 3082·2 and Cu 3073·8 (0·5 to 28); Fe 3075·9 and Cu 3073·8 (0·3 to 2); Ca 3158·9 and Cu 3273·9 (3 to 45); Si 2435·2 and Cu 2392·7 (3 to 48); Mg 2783·0 and Cu 2824·4 (0·5 to 15); Mn 2933·1 and Cu 2882·9 (6 to 30); CaF_2 6064·4 and Cu 5700 (2 to 30). The calibration curves are constructed with the co-ordinates ΔS and log C. The total content of Ca is first determined, then the concn. of CaF_2. The spectral analytical results agree well with those of chemical analysis.

C. D. KOPKIN

1534. **Determination of the total carbon content of clays and like materials, by oxidation with a solution of chromium trioxide.** W. Schmied and H. Steiner. *Glas-Email-Keramo-Tech.*, 1957, **8** (6), 215–221.—A description is given of an apparatus in which a sample of 1 to 2 g is heated under reflux with a 70% soln. of H_2SO_4 containing 2 g of $K_2Cr_2O_7$. Any hydrocarbon gases that collect with the CO_2 are oxidised by sparking with O before the total vol. of CO_2 is determined by absorption in alkali.

J. A. SUGDEN

See also Abstracts—**1429**, Precipitation separations of iron from aluminium, and nickel from cobalt. **1435**, Flame-photometric determination of sodium and potassium. Serial micro-analysis of structural constituents of steel. Spectrographic analysis of steel. Spectrographic analysis of slags. Spectrochemical analysis of grey cast-iron. Determination of selenium in pyrites. Absorption spectra of H_2O - D_2O mixtures. **1436**, High-precision spectrophotometric analysis. **1573**, Determination of silica, iron and aluminium in black coal. **1593**, Determination of manganese in tissue extracts. **1595**, Determination of nickel in biological materials. **1661**, Determination of lead.

3.—ORGANIC ANALYSIS

1535. **Classification of organic compounds based on behaviour of a solvochromic and thermochromic indicator system.** S. Soloway and P. Rosen (City Coll., New York, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1820–1823.—Organic compounds are classified according to their effectiveness in promoting or inhibiting chelation between ferric chloride and *n*-propyl gallate (**I**). The indicator system comprises $FeCl_3$, **I** and *o*-chloroaniline; the soln. may be blue or yellow throughout the entire temp. range of the liquid state, or they may exhibit a reversible thermochromic transition from blue to yellow. The solvent and temp. control the extent of chelation between the indicator components. This classification used in conjunction with others based on solubility, elementary analysis and acid - base indicators, enabled 19 out of 28 types of compounds to be identified.

G. P. COOK

1536. **The micro-determination of carbon and hydrogen in compounds containing alkali and alkaline-earth metals.** E. Kissa (Jackson Lab., Res. Div., E. I. du Pont de Nemours & Co., Inc., Wilmington, Del., U.S.A.). *Microchem. J.*, 1957, **1** (2), 203–207.—Tungsten trioxide is advocated as a tube-filling when the combustion leads to the formation of stable carbonates. It melts at 1473° and does not vaporise till 1750°. It neither spatters nor attacks the combustion tube, nor exhausts the silver gauze. The platinum boat is easily cleaned, and WO_3 is less hygroscopic than V_2O_5. The combustion is carried out at 900°, with a 6 to 8-fold weight of WO_3. Figures for C and H, with the use of both WO_3 and V_2O_5, are given for Na oxalate, citrate and acetate, K H phthalate and Ba acetate. They show good reproducibility and close agreement between results obtained by the two reagents.

R. E. ESSERY

1537. **Simultaneous micro-determination of carbon, hydrogen and alkali metal (lithium, sodium or potassium).** A. S. Zabrodina and V. P. Miroshina (Moscow Univ.). *Vestn. Moskov. Univ.*, 1957, (2), 195–198.—Combustion in O of the sample mixed with finely ground quartz in an empty tube prevents absorption of CO_2 by the alkali metal and permits C and H to be determined by the usual method, and the alkali metal to be determined from the difference in wt. of the sample and container before and after the combustion.

G. S. SMITH

1538. **Determination of carbon, hydrogen and nitrogen in organoboron compounds.** P. Arthur, R. Annino and W. P. Donahoo (Oklahoma State Univ., Stillwater, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1852–1854.—When applied to the determination of organoboron compounds, conventional

combustion techniques yield low results. By employing quartz combustion-tubes and by heating to between 1000° and 1100° in the unpacked section around the combustion boat, both C and H were determined to within about ± 0·2% and ± 4%, respectively. Determinations of N in acetanilide - BF_3 were improved by restricting the sample sizes to 7 mg or less or by employing a longer combustion tube with two successive packings. G. P. Cook

1539. **Observations on nitrogen determination. I. Examination of samples giving erroneous results.** Satoshi Mizukami, Keikichi Miyahara and Hiroko Nakai (Res. Lab., Shionogi & Co. Ltd., Imafuku, Amagasaki). *J. Pharm. Soc. Japan*, 1957, **77** (3), 312–316.—In the Dumas method of nitrogen determination, incomplete combustion occasionally results when the temp. of the fixed furnace is <750°, since the sample vapour then passes into the nitrometer without being oxidised. Compounds containing ring-nitrogen are apt to remain unburnt in the sample boat, but most of them can be burnt by mixing with CuO and keeping the sample-heater at 850° to 900°. Some compounds [*e.g.*, pyridoxine and di-(4-amino-2-methyl-5-pyrimidinylmethyl) disulphide] require the addition of Co_2O_3 to the sample boat.

II. Correction of the nitrometer reading and modification of nitrometer. Satoshi Mizukami and Keikichi Miyahara. *Ibid.*, 1957, **77** (3), 316–318.—The contribution of the capillary effect to the inside pressure of the nitrometer is ≃0·6 mm (Hg) for the milligram scale and ≃6 mm (Hg) for the decimilligram scale. A nozzle-type inlet covered with thin rubber or vinyl chloride sheet appears to be superior to a ground-joint one. K. Saito

1540. **Micro-determination of primary amino groups with nitrosyl bromide.** G. Kainz, H. Huber and F. Kasler (II. Chem. Inst., Univ. Vienna). *Mikrochim. Acta*, 1957, (5), 744–750 (in German).—High results in the Van Slyke determination of primary amino groups with HNO_2 (*cf.* Kainz and Schöller, *Anal. Abstr.*, 1956, **3**, 741) are avoided by the use of nitrosyl bromide reagent instead of HNO_2 soln. in the apparatus previously described (Kainz, *Mikrochim. Acta*, 1953, 349). The reagent is prepared by dissolving $NaNO_2$ (0·2 g) in a mixture of glacial acetic acid (1·5 ml) and Br (0·3 ml). Interfering groups are brominated before the determination. Low results are still obtained with some compounds. A. R. Rogers

1541. **Volumetric micro-determination of halogens in organic substances.** M. Ionescu and I. Goia (Inst. Chim., Acad. R.P.R., Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1957, **8** (1–2), 173–178.—The original micro-method of Irimescu and Chirnoagă (*Z. anal. Chem.*, 1942, **125**, 32), in which the organic chloride is treated with metallic sodium in ethanol, is modified. *iso*Amyl alcohol replaces ethanol; nitrobenzene is added before addition of excess of $AgNO_3$; and 0·02 *N* soln. are used instead of 0·01 *N*. If the organic material contains several halogen atoms, some of which are loosely bound, dissolution in a dil. soln. of Na amyloxide is recommended. Details of the method are given. H. Sher

1542. **Simultaneous micro-determination of fluorine, carbon and hydrogen in organic compounds.** M. O. Korshun, N. E. Gel'man and K. I. Glazova. *Dokl. Akad. Nauk, SSSR*, 1956, **111** (6), 1255–1256; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,900.—The earlier recommended method (*Anal. Abstr.*, 1954, **1**, 1534) for the simultaneous determination of F, C and H, based on the ability of MgO to trap F quant. as MgF_2, gives inconsistent results for F because of the unfavourable conversion factor and the inconstancy of the weight of the quartz tube containing MgO. In the present method for determining F, the property of MgF_2 to decompose under the action of superheated steam with the formation of HF and MgO is used. Ignite the substance (4 to 10 mg) in a tube in a current of O, passing the gas through a layer (15 to 18 cm) of granulated MgO ignited to 1000° in a quartz tube with a sintered bottom. After the ignition, submit the tube with the MgO to a temp. of 1000° and a rate of flow of steam of 0·5 ml of condensate per min. Determine F in the condensate by titration with alkali or $Th(NO_3)_4$ soln. if the substance contains P, Si and B as well as C, H, O, N and F, or with $Th(NO_3)_4$ soln. if other halogens and S are present. The time for one determination of H, C and F is 1·5 hr. C. D. Kopkin

1543. **The semi-micro determination of phosphorus in fluorinated organic compounds.** T. R. F. W. Fennell, M. W. Roberts and J. R. Webb (Royal Aircraft Estab., Farnborough, England). *Analyst*, 1957, **82**, 639–643.—Wet oxidation with HNO_3 and H_2SO_4 as a preliminary procedure in the determination of P in fluorinated organic compounds is not universally applicable, but decomposition by fusion with Na_2O_2 followed by pptn. of quinoline molybdophosphate is reliable, rapid and accurate. The sample (≡ 2 to 3 mg of P) is mixed in a micro-bomb with enough sucrose to make the organic matter up to 50 mg, KNO_3 (50 mg) and Na_2O_2 (1 g). After reaction, the contents of the bomb are leached according to a specified procedure, the leachings being run into platinum vessels in which they are acidified (to a phenolphthalein - thymol blue indicator), evaporated to low bulk, treated with a soln. of H_3BO_3 (to prevent action of F on glass) and filtered into a glass flask. The filtrate is heated to b.p. with sodium molybdate soln. and conc. HCl, and quinoline hydrochloride soln. is added slowly. The collected ppt., washed free from acid, is dissolved in standard NaOH soln. and titrated with 0·5 *N* HCl, the indicator being a mixture of phenolphthalein and thymol blue in ethanol. Results are within ±0·15% absolute. A. O. Jones

1544. **Determination of unsaturation by near-infra-red spectrophotometry.** R. F. Goddu (Hercules Powder Co., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1790–1794.—Isolated single double-bonds of several types can readily be detected and determined in the near-infra-red region of the spectrum. Terminal methylene groups may be determined at 1·62 or 2·1 μ with a sensitivity within 0·01% and a precision within ±2%. Cis-double-bonds may be determined at 2·14 μ with a sensitivity of ≃1%. Data for about 30 compounds are listed. G. P. Cook

1545. **The micro-determination of alkoxyl groups.** A. S. Inglis (Org. Chem. Dept., Univ. Liverpool, England). *Mikrochim. Acta*, 1957, (5), 677–688 (in English).—An improved apparatus and method are described. A simple modification may be used for liquids such as anisole that need a double distillation. The reaction of the sample with HI in the presence of phenol is usually very rapid at the boiling temp. and depends on the configuration of the molecule

rather than on the number of alkoxyl groups present. An aq. soln. containing $Na_2S_2O_3$ and $CdSO_4$ is an excellent scrubbing medium. A high rate of flow of CO_2 (12 to 15 ml per min.) is essential for accurate results. A. R. ROGERS

1546. **Formation of polyglycerols by etherification of glycerol.** E. Mareš (Res. Inst. of Oils and Fats, Ústí nad Labem, Czechoslovakia). *Průmysl Potravin*, 1957, **8** (3), 147–151.—The degree of etherification of glycerol can be controlled very simply by measuring the refractive index. J. ZÝKA

1547. **Ultra-micro determination of fructose with *p*-anisyl blue ditetrazolium chloride.** E. M. Livingston (Brooklyn Coll., N.Y., U.S.A.). *Microchem. J.*, 1957, **1** (2), 265–268.—The blue colour developed on heating fructose with the reagent is measured. On the day of analysis mix 1 ml of 1% *p*-anisyl blue ditetrazolium chloride in 50% ethanol with 3 ml of 0·3 *M* NaOH. To 0·5 ml of fructose soln. (containing up to 50 μg) in a graduated test-tube add 0·5 ml of the reagent mixture, shake, stopper with a cold-finger condenser to provide reflux and heat to boiling-point as rapidly as possible with a large flame from a micro-burner. When the boiling-point is reached, lower the flame to maintain gentle boiling for 73 sec., then cool in water for 2 min. Dilute with dioxan, but do not make up to 12·5 ml until room temp. is reached. Measure the extinction at 615 mμ, with dioxan as blank. Beer's law is not followed, but there is a linear relationship between extinction and concn. up to 2·5 μg per ml. The blue colour decomposes with heating so that, with replicate results, owing to slight differences in the times required to reach boiling-point, the highest result should be accepted.

R. E. ESSERY

1548. **Micro-determination of fructose with the phenol reagent.** E. M. Livingston, R. K. Maurmeyer and A. Worthman (Brooklyn Coll., N.Y., U.S.A.). *Microchem. J.*, 1957, **1** (2), 261–264.—The green colour produced on heating fructose with phenol and conc. H_2SO_4 is measured spectrophotometrically. The fructose soln. (containing 0·5 to 3 mg) is placed in a specimen tube and evaporated to dryness *in vacuo* over H_2SO_4. Phenol soln. (10·5% in glacial acetic acid) (0·02 ml) and 0·06 ml of H_2SO_4 soln. (1 vol. of conc. acid + 9 vol. of glacial acetic acid) are added, and the tube is heated in a boiling-water bath for 9 $\pm$ 1 min., with shaking, then cooled under the tap for 1 min. The mixture is made up to 25 ml with glacial acetic acid, and the extinction is read at 326 mμ, with glacial acetic acid as a blank. Beer's law is followed with pure fructose, but in the presence of other sugars a calibration curve is necessary. Water in any form leads to low results. R. E. ESSERY

1549. **Infra-red micro-techniques for identification of carbohydrates and other organic compounds.** F. E. Resnik, L. S. Harrow, J. C. Holmes, M. E. Bill and F. L. Greene (Philip Morris, Inc., Richmond, Va., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1874–1877.—The micro-techniques described produce good i. r. spectra without extensive instrument modification or expensive accessories. The main features of the techniques are the use of a KBr micro pellet, which extends the range of detection to microgram quantities without the use of beam-condensing lenses, and the adaptation of the lyophilisation technique (freeze drying). The usefulness of the method is illustrated by a study of the carbohydrates in tobacco and it may be applied to both water-soluble and water-insoluble materials such as may be found in natural products. The use of an internal standard allows practical precision in the quant. determination of micro quantities of materials. G. P. COOK

1550. **Polarographic determination of the methylene blue number of regenerated celluloses of high carboxyl content by the suppression of oxygen maxima.** I. Rusznák, I. Králik and K. Fukker (Res. Inst. for the Textile Ind., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 59–71.—Details are given for the determination of the methylene blue numbers of regenerated celluloses and conversion of these values to carboxyl content. A stoicheiometric ratio of two carboxyl groups per methylene blue molecule was established. Results on samples having carboxyl contents ranging from 0·4% to 22·4% were higher than those given by the Ca acetate method (*J. Amer. Chem. Soc.*, 1942, **64**, 121; *Ibid.*, 127; *Trans. Faraday Soc.*, 1942, **38**, 209). G. H. FOXLEY

1551. **New colour reaction of aldehydes.** T. Urbanskiĭ. *Bull. Acad. Polon. Sci.*, 1956, **4** (9), 589–590; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,920.—In the reaction of aldehydes (in dil. aq. soln.) with H-acid (**I**) or its sodium salt in H_2SO_4 medium, a yellow colour is formed. The reagent is prepared by mixing conc. H_2SO_4 (100 ml) and water (33·3 ml) and adding 0·70 g of **I**, to give a 0·5% soln. in 80% H_2SO_4. Add a few ml of reagent soln. to $\simeq$1 ml of the soln. to be tested. In the presence of a considerable quantity of aldehyde an immediate yellow or orange colour is formed; with a smaller amount of aldehyde the colour develops on warming. The detectable minimum for formaldehyde, benzaldehyde, cinnamaldehyde and furfuraldehyde are, respectively, 0·01, 0·01, 0·002 and 0·002%. The analogous reaction for ketones is less sensitive. C. D. KOPKIN

1552. **Organic chemical microscopy. II. Anilides as qualitative organic derivatives of carboxylic acids.** R. E. Dunbar and C. C. Moore (N. Dakota State Coll., Fargo, U.S.A.). *Microchem. J.*, 1957, **1** (2), 209–222.—The anilides are prepared by suitable methods (given), recryst. from dilute aq. ethanol, and examined microscopically under a low power. Photomicrographs are included of anilides of 8 liquid and 21 solid acids, 4 of the solids being dicarboxylic. In some instances polymorphic forms appear, depending on the precise conditions of crystallisation, and warning is given against confusion of a possible derivative with an initial solid acid, to be avoided by comparison with crystals of the acid obtained from a suitable solvent. It is suggested that the microscopy of anilides forms a useful addition to the tests of identity of carboxylic acids. R. E. ESSERY

1553. **Colour reaction of organic compounds with dimethylglyoxime. III. Detection of hydrazine and some nitrogenous organic compounds.** Seiichi Okuma and Yasumasa Kido (National Hyg. Lab., Tamagawa-yoga, Setagaya-ku, Tokyo). *J. Pharm. Soc. Japan*, 1956, **76** (8), 894–896.—Colour reactions of dimethylglyoxime with hydrazine and acid hydrazides after heating with KCNO are described. Certain barbiturates after heating with NaOH soln. give a red colour with a dimethylglyoxime reagent on heating with thiosemicarbazide. This test is also given by guanidine, aminoguanidine, arginine and creatinine. K. SAITO

1554. **The u.v. spectrophotometric determination of potassium ethylxanthate.** M. J. Maurice and J. L. Mulder (N. V. Onderzoekingsinst. "Research," AKU, Arnhem, Netherlands). *Mikrochim. Acta*, 1957, (5), 661–667 (in English).—To determine small amounts of CS_2 (as K ethylxanthate), absorb the sample (containing 100 to 600 μg of CS_2) in 0·1 *N* ethanolic KOH (50 ml), dilute with H_2O to 100 ml and compare the extinction at 301 mμ with that of a 0·05 *N* soln. of KOH in 50% ethanol. The molar extinction coeff. is $\simeq$ 17,820. The standard deviation of a determination is $\simeq \pm$ 2·2 μg (estimate based on 32 assays). A. R. ROGERS

1555. **Micro-determination of boron in organoboron compounds.** P. Arthur and W. P. Donahoo (Oklahoma Agric. Mech. Coll., Stillwater). *U.S. Atomic Energy Comm.*, Rep. CCC–1024–TR–221, 1957, 22 pp.—A procedure has been developed for the combustion of micro samples of organoboron compounds. The B_2O_6 formed by the combustion is quant. removed by refluxing water in the tube, and the resulting $B(OH)_3$ soln. is titrated with NaOH, with mannitol as a complexing agent, with an initial pH (before adding mannitol) of 5·4 and an end-point pH of 8·4. Best results are obtained when the content of B of the soln. is between 0·8 and 1·5 mg. NUCL. SCI. ABSTR.

1556. **Determination of dimethoxyborane in methyl borate solutions.** A. J. Krol, L. B. Eddy, D. R. Mackey and A. E. Weber (Callery Chem. Co., Pa.). *U.S. Atomic Energy Comm.*, Rep. CCC–1024–TR–239, 1957, 12 pp.—A titrimetric method has been developed for the determination of hydrolysable H in $HB(OMe)_2$, based on the reaction of hydridic hydrogen with iodine and a subsequent titration of excess of iodine with thiosulphate. The method was found to be directly applicable to $HB(OMe)_2$ in $B(OMe)_3$ soln. in the concn. range 0 to 15% of $HB(OMe)_2$. Higher concn. of $HB(OMe)_2$ could be determined after preliminary dilution of the sample with $B(OMe)_3$. Solutions of iodine in either toluene or *iso*propyl alcohol were suitable; *iso*propyl alcohol soln. were preferred, because the back-titration of the excess of iodine could be carried out in a single liquid phase. Instrumental analysis based on the measurement of refractive index appeared to have a low sensitivity, $\simeq$ 1% of $HB(OMe)_2$ being detectable. Analyses based on the dielectric-constant measurement by means of a chemical oscillometer were found to be somewhat more sensitive, $\simeq$ 0·5% of $HB(OMe)_2$ being detectable. Infra-red analysis of soln. is indicated as a relatively sensitive method. NUCL. SCI. ABSTR.

1557. **Determination of hydroxylic impurities in methyl borate by infra-red spectrophotometry.** D. R. Mackey, J. W. Brogan, H. J. Birch and A. E. Weber (Callery Chem. Co., Pa.). *U.S. Atomic Energy Comm.*, Rep. CCC–1024–TR–232, 1957, 13 pp.—Among the major impurities in methyl borate are those containing hydroxyl groups, particularly methanol and boric acid. The concn. of hydroxyl groups can be estimated with a precision of 0·04 milli-equiv. per ml in the range of 0 to 0·5 milli-equiv. per ml by quant. i.r. analysis. The methods used for establishing working curves are the base-line method and area methods, in particular a new method, the relative integrated absorbance. NUCL. SCI. ABSTR.

1558. **Determination of coumarin by the alkaline hydroxide method.** N. A. Valyashko and É. G. Berdichevskiĭ (Kharkov Pharm. Inst.). *Zhur. Obshch. Khim.*, 1957, **27** (8), 2302–2303.—The sample containing 0·1 to 0·3 g of coumarin is dissolved in 10 ml of ethanol. The soln. is shaken energetically with 0·1 g of yellow HgO and then treated at room temp. for 15 min. with 10 ml of 0·5 *N* ethanolic NaOH. After dilution to 100 ml and filtration, the excess of NaOH in a 25-ml portion is titrated. G. S. SMITH

1559. **Analytical separation of salicylic acid from sulphosalicylic acid.** M. Vietti-Michelina (Inst. für Warenkunde, Turin Univ., Italy). *Z. anal. Chem.*, 1957, **157** (5), 346–347.—Paper-chromatographic and -electrophoretic methods are described. R_F values given show salicylic acid to move more quickly than sulphosalicylic acid; in an acid medium (butanol, acetic acid and H_2O) the values are 0·75 and 0·05 with descending technique, and 1·0 and 0·4 with ascending technique; in an alkaline medium (butanol and aq. NH_3), 0·44 and 0·0 (ascending), and 0·41 and 0·0 (descending). Paper electrophoresis gives more rapid separation, sulphosalicylic acid moving the faster, in the ratio 16 cm in 2 hr. to 5 cm in 2 hr. in 5% aq. NH_3, and in the ratio 6·2 cm in 1 hr. or 3·8 cm in 0·5 hr. to 2·5 cm in 1 hr. or 1 cm in 0·5 hr. in acid. By this method 5 μg of each acid in a mixture may be quickly separated. E. G. CUMMINS

1560. **Quantitative determination of methyl and ethyl salicylates.** A. N. Gengrinovich and Ya. K. Kadȳrov (Tashkent Pharm. Inst.). *Aptechnoe Delo*, 1957, **6** (2), 68–69.—Methyl or ethyl salicylate is saponified by aq. alkali and the salicylic acid is quant. iodinated by a soln. of ICl in HCl. *Procedure*—A weighed sample (0·4 to 0·5 g) is heated under reflux for 5 min. with 25 ml of 0·5 *N* aq. KOH. The cooled soln. is neutralised to phenolphthalein with 0·5 *N* H_2SO_4 and made up to 100 ml. A 10-ml aliquot is diluted with 100 ml of water in a 250-ml stoppered flask and 20 ml of 0·1 *N* ICl is added; the flask is shaken and set aside for 2 min., 10 ml of a 10% soln. of KI is added and the liberated iodine is titrated with 0·1 *N* $Na_2S_2O_3$. E. HAYES

1561. **Oxidation of aromatic amines. I. New colour reactions of aromatic amines with hydrated manganese oxide.** M. Genchev and B. Atanasov. *Izv. Khim. Inst. Bulgar. Akad. Nauk*, 1956, **4**, 233–249; *Ref. Zhur., Khim.*, 1957, Abstr. No. 51,645.—For the identification of aromatic amines use is made of the reaction with freshly pptd. $Mn(OH)_2$ in alkaline medium, with subsequent acidification of the soln. Mix 0·1 to 0·5 g of sample with 1·5 to 2 ml of water, 0·5 ml of 1% $Mn(NO_3)_2$ soln. and 0·5 ml of 1% NaOH soln. Shake the mixture to obtain the brown ppt. of $MnO(OH)_2$ and acidify with 0·5 to 1 ml of conc. organic or dil. (1:4) mineral acid. A description is given of the characteristic reactions of aniline, methylaniline, dimethylaniline, *o*-, *m*- and *p*-toluidines, *o*-tolidine, *o*- and *p*-anisidines, *o*-, *m*- and *p*-phenylenediamines, tolylene-2:4-diamine, *p*-aminodiphenylamine, benzidine, 1- and 2-naphthylamines and diphenylamine. The sensitivities of the reactions vary from 1 μg (for *o*-anisidine) to 80 μg (for 2-naphthylamine). Methods are described for the detection of isomers of amines and of individual substances in a mixture of amines. C. D. KOPKIN

1562. **Separation of *m*- and *p*-nitrophenols by paper electrophoresis.** M. Vietti-Michelina (Inst. für Warenkunde, Turin, Italy). *Z. anal. Chem.*, 1957, **157** (4), 267–268.—The electrolyte chosen was made up from 50 ml of 0·2 *M* KH_2PO_4 and 45·2 ml of 0·2 *M* NaOH, buffered at pH 7·8.

E. G. CUMMINS

1563. **Separation of 2:4-, 2:5- and 2:6-dinitrophenols by paper electrophoresis.** M. Vietti-Michelina (Inst. für Warenkunde, Turin, Italy). *Z. anal. Chem.*, 1957, **157** (4), 266–267.—Electrolytes such as aq. soln. of acetic acid, NaOH and HNO_3 of various concn. were tested. Best results were obtained with a buffer soln. (pH 5·3) of 0·1 *M* NaOH - 0·1 *M* Na citrate (1:4). E. G. CUMMINS

1564. **Infra-red spectra of heterocyclic compounds. I. Infra-red spectra of alkylpyridines.** Hideyo Shindo and Nobuo Ikekawa (Sankyo Co. Ltd., Tokyo). *Pharm. Bull., Japan*, 1956, **4**, 192–197.—The i.r. spectra of 8 ethyl- and 19 methyl-pyridines were determined.

II. Infra-red spectra of methylquinolines. Hideyo Shindo and Shinzo Tamura. *Ibid.*, 1956, **4**, 292–297.—The i.r. spectra of quinoline, *iso*quinoline and their monomethyl deriv. are recorded. These compounds give highly characteristic bands in the finger-print region of the spectrum which seem to be useful in the analysis of tar bases. N. E.

1565. **Azeotropic distillation as a method for the control of drugs. III. Determination of constituents of the so-called "picoline" fraction.** B. Melichar (Inst. Pharm. Chem., Masaryk Univ., Brno, Czechoslovakia). *Československ. Farm.*, 1957, **6** (4), 209–212.—The method is based on the formation of homoazeotropic mixtures of the compounds with water. The characteristic boiling-point depressions are 4·70° for α-picoline, 2·96° for β-picoline, 2·56° for γ-picoline and 3·88° for 2:6-lutidine. The amount of the compound is indicated by the vol. of the distillate. Only 0·5 to 1·0 ml of sample is required.

J. VOLKE

1566. **Application of titration in non-aqueous solutions. I. Determination of 5-acetamidomethyl-4-amino-2-methylpyrimidine in glacial acetic acid.** Satoshi Mizukami and Eizo Hirai (Res. Lab., Shionogi & Co. Ltd., Imafuku, Amagasaki). *J. Pharm. Soc. Japan*, 1957, **77** (2), 165–167.—5-Acetamidomethyl-4-amino-2-methylpyrimidine (**I**) (≃200 mg) behaves as a mono-acidic base in glacial acetic acid (**II**) (30 ml) and can be titrated potentiometrically with 0·1 *N* $HClO_4$. A mixture of methyl violet (0·1% in chlorobenzene) and bromophenol blue (0·4% in **II**) (1 + 1, v/v) is used as the indicator, the colour change being from reddish-amber to blue. 4-Amino-5-ethoxymethyl-2-methylpyrimidine and Na acetate also react with $HClO_4$; the former compound is extracted with chlorobenzene (**I** is insol.) and titrated with $HClO_4$, with methyl violet as indicator, and the Na acetate is converted by ion exchange into acetic acid, which is titrated with NaOH in aq. soln. These results are deducted from the overall titre in **II**.

K. SAITO

1567. **Determination of mononitrothiophen and dinitrothiophen in nitrobenzene.** W. Leibmann and J. T. Woods (American Cyanamid Co., Bound Brook, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1845–1846.—Dinitrothiophen is determined in nitrobenzene by its colour reaction with ethanol and NaOH; the extinction is measured at 540 mμ and both the 2:4- and the 2:5-isomers give the same colour. Mononitrothiophen is quantitatively nitrated to the dinitro compound, which is determined by the same method; trinitrobenzene interferes. The standard deviation is within ±3% (relative) over the range of 5 to 500 p.p.m.

G. P. COOK

1568. **Analysis of aromatic sulphonation reaction mixtures.** W. H. Houff, D. R. Christie and R. H. Beaumont (F. C. Huyck and Sons, Kenwood Mills, Rensselaer, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1866–1868.—Aromatic sulphonation mixtures containing H_2SO_4 and Na_2SO_4 are analysed by a three-stage procedure. After pptn. of SO_4^{2-} with $Ba(OH)_2$ and passage of the filtrate through a cation-exchange resin, the regenerated sulphonic acid is determined by titration with NaOH. The total acidity is determined by direct titration with NaOH, and the total SO_4^{2-} by the gravimetric pptn. procedure; these determinations in conjunction with the sulphonic acid determination enable the H_2SO_4 and Na_2SO_4 contents to be calculated. In an alternative procedure the total acidity and total anion content are determined. Recoveries from synthetic mixtures and commercially available sulphonic acids were ≃ 100% for all procedures.

G. P. COOK

1569. **Analysis of toxic phosphorus compounds.** D. N. Kramer and R. M. Gamson. *Anal. Chem.*, 1957, **29** (12), 21A–28A.—A survey of the general and specific analytical methods available is presented. Specific procedures are summarised in a table which gives the approximate sensitivity and the interferences. The methods discussed include colorimetry, i. r. and u. v. spectrophotometry, polarography, bio-assay, enzymic, conductimetric, electrometric and chromatographic techniques.

G. P. COOK

1570. **Distribution of normal paraffins and separation of saturated hydrocarbons from recent marine sediments.** E. D. Evans, G. S. Kenny, W. G. Meinschein and E. E. Bray (Magnolia Petroleum Co., Dallas, Tex., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1858–1861.—Alumina and silica gel chromatographic separations and mass spectrometry were used to study these saturated hydrocarbons. The normal paraffins have higher concn. of C_{2n+1} molecules than of their C_{2n} homologues. Mass-spectrometric analysis of the chromatographic fractions established the efficiency of the separation of naphthenic and paraffinic hydrocarbons on alumina columns at different adsorbent to sample ratios, and showed that normal paraffin mixtures are separated on a mol. wt. basis on alumina at certain adsorbent to sample ratios.

G. P. COOK

1571. **Examination of mineral oils. Determination of salt content.** *Erdöl u. Kohle*, 1957, **10** (5), 315–316.—A provisional standard method for the estimation of inorganic chloride in oils is described. The sample is mixed with benzene, water and acetone in a special extraction apparatus, allowed to boil for 1 hr., then cooled, and the aqueous phase separated. The sodium chloride is then titrated by the Volhard method. A reproducibility of 0·004% is claimed. If sulphides are present a small modification is necessary. P. S. STROSS

1572. **Methods for the analysis and testing of coal and coke. 1. Total moisture of coal.** British Standards Institution (2, Park St., London). B.S. 1016: Part 1:1957, 15 pp.—Methods for the

determination of the moisture in coal "as sampled" are described. In the distillation method for the half-inch sample, toluene has been substituted for the petroleum distillate of boiling range from 150° to 180° which can give high results.

O. M. WHITTON

1573. **Spectrographic determination of silica, iron and aluminium in black coal.** M. F. Kessler and F. Valeška (Phys. Chem. Lab., Res. Inst. Fuels, Prague, Czechoslovakia). *Paliva*, 1957, **37** (5), 158–160.—A method is described for the direct determination of the principal inorganic components of coal, without previous ignition of the sample.

J. ZÝKA

1574. **Carbon - hydrogen analysis of coke on catalysts.** S. G. Hindin, J. K. Lee and S. W. Weller (Houdry Process Corp., Marcus Hook, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1850–1852.—The coked catalyst is heated with a known, limited, volume of O, slightly in excess of that required for complete combustion. The water and CO_2 formed by combustion are determined from the v.p. in the system, after each combustion product has been separately frozen by a suitable low-temp. bath. For a series of samples having coke levels in the range 0·2 to 4% by wt., the coeff. of variation of the hydrogen determination was $\simeq \pm 3{\cdot}7\%$; the coeff. of variation of the carbon determination was $\pm 2{\cdot}0\%$.

G. P. COOK

1575. **The determination of naphthalene and benzole in coke-oven gas.** I. Moulson (British Coke Res. Ass., 74 Grosvenor St., London). *Gas World*, 1957, **145**, 54–60 (Suppl.).—From 0·75 to 2 g of naphthalene per 100 cu. ft. of washed gas can be determined as picrate with 0·7 to 0·9% picric acid soln. The ppt. is washed with aq. picric acid ($\ll 0{\cdot}02\ N$), transferred to a beaker with acetone ($\simeq 5$ ml) and the soln. is titrated at 15° with 0·1*N* NaOH (bromothymol blue indicator). Glacial acetic acid (50 ml) is added, followed by Wijs' reagent (10 ml) and, after being set aside for 20 min. in darkness, 10% KI soln. (10 ml). The mixture is then titrated with 0·1 *N* $Na_2S_2O_3$ and the indene correction is calculated. The content of naphthalene is slightly higher for the Ruhrgas method of collection and pptn. (at $\simeq 0°$, rate of flow $\gg 3$ cu. ft. per hr.) than for the B.C.R.A. method (thermostatic control at 10° to 15°, rate of flow $\gg 12$ cu. ft. per hr.). The latter method is very suitable for determining low concn. of naphthalene; one test can be made in $\simeq 24$ hr. The B.C.R.A. absorption train described and illustrated can recover 20 ml of benzole for fractionation through a Dufton column (*cf.* Gooderham, *Brit. Abstr. C*, 1944, 68). Water in the stripped gas (rate of flow 15 to 25 cu. ft. per hr.) is removed by freezing and by Anhydrone, whilst the benzole is condensed by means of acetone and CO_2 at $\simeq -80°$. One test can be made in 10 to 12 hr.

W. J. BAKER

1576. **Experiences in the practical application of paper chromatography in the soap field.** K. Röth (Feinseifenwerk Walter Rau & Co., Stuttgart-Möhringen). *Fette, Seif., Anstrichmitt.*, 1957, **59** (6), 418.—A method for separating fatty acids on non-impregnated paper is described. A sample of the mixed acids, dissolved in toluene, is deposited on S. & S. 2043b or similar paper. When the spot has dried, the paper is wound on a glass rod at right angles to the direction of development; the rod is dipped about 1 to 2 mm into a "barrier" liquid such as decalin, the excess of liquid being wiped off with filter-paper when the rod is withdrawn. The paper is then developed by the ascending technique with acetic acid - methanol - water (74:15:11) for 3 hr. Stearic acid and its higher homologues remain at the starting-point, but oleic, myristic, lauric and caprylic acids separate, forming spots that can be easily distinguished. The "barrier" broadens and moves with the solvent during the development.

E. HAYES

1577. **New analytical method for the detection of thiols in cold-wave preparations.** G. T. Walker, F. M. Freeman and F. Hirsch. *Seifen-Öle*, 1956, **82**, 161–163.—The colour reaction with ninhydrin in slightly alkaline medium can be used to detect thiols (primary, secondary and tertiary); the colour is slightly pink at pH 4, reaches a deep-red max. at pH 9 and, even in the absence of thiols, becomes intensely blue at high alkalinity; the colour is stable under atmospheric conditions. Thioglycollic acid (70 p.p.m.) can be detected.

CHEM. ABSTR.

1578. **Selective solvents for analysing textile fibre mixtures.** S. S. Praeger (Div. of Bedding Lab., New York State Dept. of Labor, New York). *Amer. Dyest. Rep.*, 1957, **46** (14), 497–498.—A chart of solubilities of both natural and synthetic fibres in various solvents is given. Its application in analysing fibre mixtures is illustrated and discussed.

S.C.I. ABSTR.

1579. **Chromatographic method for identification of amino aldehyde finishes on textiles.** B. Norwick and E. Bierig (Beaunit Mills, Inc., New York, N.Y., U.S.A.). *Amer. Dyest. Rep.*, 1957, **46** (9), 314–315.—Descending paper chromatography provides a simplified technique for the identification of the amino constituents hydrolysed from aldehyde - amine finishes on textiles with 0·1 *N* HCl. The developing solution is *n*-butanol - ethanol - H_2O (3:1:1, by vol.), and 3 hr. is a suitable development time. The spots are made visible by treatment with formaldehyde, followed by curing and staining with Scarlet 4RA (C.I. 185).

E. S. LANE

1580. **Determination of xanthate sulphur in viscose.** J. P. Dux and L. H. Phifer (American Viscose Corp., Marcus Hook, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1842–1845.—Undiluted viscose is forced through a specially designed anion-exchange column, which removes the sulphur compounds other than cellulose xanthate. A portion of the eluate is weighed and diluted and the xanthate content of this soln. is measured by u.v. absorptiometry at 303 mμ. The method is not absolute but must be calibrated in terms of the gravimetric sulphate procedure. The coeff. of variation is $\pm 2{\cdot}8\%$ from 19 determinations.

G. P. COOK

1581. **A simple technique for use with infra-red spectroscopy to study the curing of epoxy resins.** C. E. Feazel and E. A. Verchot (Southern Res. Inst., Birmingham, Alabama, U.S.A.). *J. Polym. Sci.*, 1957, **25** (110), 351–353.—In the technique described, neither sacrifice of rock-salt plates nor use of special grinding equipment is necessary. The mixture of epoxy resin and curing agent is spread between two pellets of pure KBr made by the pressed disc technique. The thickness of the

resin film is kept constant as the resin is cured by heating at 60° ± 1°, during which the i.r. spectrum of the film is measured. The technique has been used to compare the activities of various diamines as curing agents. B. J. WALBY

1582. **Dielectric constant and its value for the analysis of paint raw materials.** P. Baur (Herbig-Haarhaus A.-G., Köln-Bickendorf, Germany). *Farbe u. Lack*, 1957, **63**, 284–289.—An immersion method of dielectric-constant measurement has been developed for distinguishing anatase from rutile TiO_2. The use of such measurements to follow the oxidation and polymerisation processes of drying oils and for the control of distillation analyses are described. L. A. O'NEILL

1583. **Detection of oiticica oil in coatings.** G. G. Esposito and M. H. Swann (Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1861–1862.—Oiticica oil can be detected in coating materials by the red colour produced with *m*-dinitrobenzene, which reacts with the characteristic carbonyl group of licanic acid, the principal acid constituent of the oil. G. P. COOK

1584. **Determination of free sulphur in rubber containing sulphur-containing accelerators.** E. P. Kheraskova, N. A. Okhapkina and V. N. Provorov (Sci. Res. Inst. for Rubber Articles in General Use). *Zavod. Lab.*, 1957, **23** (7), 798–800.—The normal sulphite method for determining free S in rubber (*cf.* ASTM standard 1952) is unsatisfactory in the presence of tetramethylthiuram disulphide and other S-containing accelerators. The use of activated C is recommended. The sample (2 g) of rubber is boiled for 2 hr. with 100 ml of 5% Na_2SO_3, and the cooled soln. is treated with 3 g of activated C and periodically shaken during 30 min. The soln. is then filtered and the filter is washed with water, 5 ml of 40% formaldehyde is added to the filtrate, followed after 5 min. by 20% acetic acid soln. to acidify the soln., which is then titrated with 0·1 *N* iodine in the presence of starch. The C must be tested under similar conditions; the amount of 0·1 *N* iodine required should not exceed 0·1 ml. G. S. SMITH

1585. **Determination of mercaptobenzothiazole (MBT) in technical Captax and Altax.** G. Blöckinger (Dept. Chem., Univ., Bratislava). *Chem. Zvesti*, 1957, **11** (6), 340–346.—An iodimetric method is described which avoids the influence of other materials such as thiocarbanilide, triphenylguanidine, resins and dibenzothiazolyl disulphide which are formed in the production process. The interference of thiocarbanilide is avoided by carrying out the titration quickly at high dilution. N. E.

1586. **The application of diphenylamine and related compounds to spot-tests for nitrate and nitramine explosives.** T. M. Finnie and H. J. Yallop (Armament Res. and Development Estab., Fort Halstead, Kent, England). *Analyst*, 1957, **82**, 653.—In the use of diphenylamine, diphenylbenzidine, nitrodiphenylamine and brucine in H_2SO_4 soln. for the testing of nitrates and nitramines the concn. of the acid is important. The nitramines examined were *cyclo*trimethylenetrinitramine (RDX), *cyclo*tetramethylenetetranitramine (HMX), picrite (nitroguanidine) and tetryl (2:4:6-trinitrophenylmethylnitramine). Two explosives, *viz*, pentaerythritol tetranitrate (PETN) and urea nitrate, were also used for comparison. A drop of the reagent was applied to the dry test substance and the colour developed at various time intervals was noted. The most satisfactory results were obtained with 80% H_2SO_4 with a 5-min. reaction period. With diphenylamine in conc. H_2SO_4, applied cold, RDX reacts in 1 min. whereas HMX requires over 5 min. With the warm reagent there is no distinction. Diphenylbenzidine and nitrodiphenylamine react similarly to diphenylamine, but the first reagent gives more reproducible results. Brucine in conc. H_2SO_4 gives an orange colour with nitramines as well as with nitrates. A. O. JONES

1587. **Infra-red quantitative analysis data.** *Anal. Chem.*, 1957, **29** (12), 1895–1896; 1958, **30** (1), 155–156.—The following data have been published under the co-operative programme between *Analytical Chemistry* and the Coblentz Society of the U.S.A. **Determination of butyl ester of stearic acid in polystyrene,** L. R. Kiley. **Determination of diethylaluminium hydride and diethylaluminium ethoxide in triethylaluminium,** R. L. Hudson. **Determination of acetylsalicylic acid, phenacetin and caffeine in mixtures,** W. H. Washburn and E. O. Krueger. **Determination of 2-bromopentane and 3-bromopentane mixtures,** F. A. Scheske and W. H. Washburn. **Determination of benzene and chlorobenzenes in chlorinated benzene,** L. R. Kiley.

Analysis of *iso*butane, *iso*butylene, 1-butene, *n*-butane and *cis*- and *trans*-2-butene, R. M. Guedin, M. C. Harvey and R. C. Wilkerson. **Determination of 1-butene, *cis*-2-butene, *trans*-2-butene, *iso*butylene and 1:3-butadiene in C_4 hydrocarbons,** L. R. Kiley. **Determination of pentan-2-ol and pentan-3-ol in mixtures,** W. H. Washburn. **Analysis of 2-methylpenten-2-al - propionaldehyde mixtures,** R. T. Scheddel and L. R. Kiley. **Determination of diethyl malonate in diethyl ethylmalonate,** W. H. Washburn. **Determination of phenol, 2-chlorophenol, 4-chlorophenol and 2:4-dichlorophenol in 4-chlorophenol,** R. T. Scheddel and L. R. Kiley.

See also Abstracts—**1434**, Precision in gas chromatography. **1435**, Fluorescence spectra of kerosines. Determination of tertiary butyl groups. Infra-red spectra of phthalocyanines. Infra-red study of organic salt solutions. Infra-red spectra of some S - O compounds. **1462**, Analysis of lithopone. **1481**, Determination of lead in paints. **1661**, Microchemical analysis of organic substances with chloropalladic acid. **1678**, Titration of organic compounds with N-bromosuccinimide. **1725**, Hydrocarbon measurements in drilling muds. **1726**, Combustion micro-analysis of volatile liquids.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1588. **Serum sodium estimation.** R. F. L. Maruna (Zentral-Lab. des Gesund.-Minist., Djakarta, Indonesia). *Clin. Chim. Acta*, 1957, **2** (6), 581–585.—Sodium is pptd. from a trichloroacetic acid filtrate of serum with magnesium uranyl acetate. The washed, vacuum-dried ppt. is dissolved in 2·5% NaOH soln. and the extinction is determined at 420 mμ. The proposed procedure is more satisfactory than the use of any of 12 other colour reactions tested. H. F. W. KIRKPATRICK

1589. **Determination of magnesium in serum and urine by the Titan yellow method.** E. Andreasen (Central Lab., Centralsygehuset, Holbaek, Denmark). *Scand. J. Clin. Lab. Invest.*, 1957, **9**, 138–143.—Serum was mixed with trichloroacetic acid and the ppt. was centrifuged off. Clear centrifugate was mixed with poly(vinyl alcohol), Titan yellow and NaOH and allowed to stand for 10 min. The extinction was then measured in a Beckman photometer at 540 mμ. The standard was $MgSO_4$ soln. instead of serum, and water replaced serum in the blank. For urine, the blank was urine treated in the same way, but with water instead of Titan yellow to compensate for the colour of the urine itself. The mean value for serum magnesium for 40 normal subjects, 20 to 40 years of age, was 1·87 milli-equiv. per litre, which is in good agreement with results of other methods.

NUTR. ABSTR.

1590. **A micro-method for the estimation of serum calcium.** S. Baar (Birmingham Accident Hosp., England). *Clin. Chim. Acta*, 1957, **2** (6), 567–575.—A stock saturated aq. soln. of Nuclear Fast Red is prepared; for use, 8 ml is mixed with *N* NaOH (5 ml) and diluted to 50 ml with H_2O. *Procedure*—To the dil. reagent (2 ml) add H_2O (0·5 ml) and the serum (0·1 ml), mix and allow to stand for 30 min. at room temp. Read at 580 mμ, against a reference soln. containing Mg (2 mg per 100 ml) treated similarly, in a narrow-band spectrophotometer. Treat various standard soln. similarly and read against a reagent blank. The content of Ca is directly proportional to the extinction for concn. up to 16 mg per 100 ml. H. F. W. KIRKPATRICK

1591. **Simple specific titration method for serum calcium.** D. N. Baron and J. L. Bell (Royal Free Hosp., London). *Clin. Chim. Acta*, 1957, **2** (4), 327–331.—The method of Tucker (*Analyst*, 1957, **82**, 284) is adapted for the direct titration of 0·1 to 1·0 ml of serum. H. F. W. KIRKPATRICK

1592. **Colorimetric estimation of calcium in serum with o-cresolphthalein complexone.** J. Stern and W. H. P. Lewis (Fountain Hosp., London). *Clin. Chim. Acta*, 1957, **2** (6), 576–580.—Calcium is pptd. from serum (0·1 ml) as oxalate and the ppt. is heated at about 500° for 30 min. The residue is treated with *N* HCl (0·1 ml), dried at 100° to 150°, heated to 200° for 5 min., and allowed to cool. The colour reagent is then added and the colour read at 575 mμ. H. F. W. KIRKPATRICK

1593. **Determination of manganese in tissue extracts by means of formaldoxime.** W. Bartley, B. M. Notton and W. C. Werkheiser (Dept. of Biochem., Univ. of Oxford). *Biochem. J.*, 1957, **67** (2), 291–295.—A simple spectrophotometric method is described for the determination of Mn in $HClO_4$ or trichloroacetic acid extracts of biological material. The Mn - formaldoxime complex has max. absorption at 450 mμ; yellow tissue constituents absorb at the same wavelength, and when large amounts are present the accuracy of the procedure is decreased. The method is applicable to the determination of 0·01 to 0·25 μmole of Mn^{2+} in 3 ml of extract with less than 5% error. The method cannot be used in the presence of Ni, and a process for the removal of Fe (which also interferes) is described. The method is applicable in the presence of large amounts of metal-complex-forming agents such as cyanide or EDTA. The method is unsuitable for the determination of endogenous tissue manganese without prior ashing of the tissue, but it works well at those levels of Mn (10^{-3} to 10^{-5} *M*) which are often used in enzyme work.

J. N. ASHLEY

1594. **A method for the determination of non-haem iron in bone marrow.** L. M. H. Kerr (Rheumatic Unit, Northern Gen. Hosp., Edinburgh, Scotland). *Biochem. J.*, 1957, **67** (4), 627–630.—The accuracy of the method is sufficient for diagnostic purposes. The content of Fe in the marrow is related to the protein content, and is determined in the homogenised marrow sample by the dipyridyl method of Ramsay and Campbell (*Biochem. J.*, 1954, **58**, 313), suitably modified for small amounts of tissue. A modified method of Lowry *et al.* (*Brit. Abstr. C*, 1952, 160) is used for the determination of protein. J. N. ASHLEY

1595. **Use of a chromatographic method in forensic chemical analysis of biological materials for the presence of nickel compounds.** L. M. Provotorova (Moscow Pharm. Inst.). *Aptechnoe Delo*, 1957, **6** (3), 28–32.—Even the natural Ni in tissues can be detected with dithio-oxamide or dimethylglyoxime, and chromatography is used with these reagents only to demonstrate the presence of Ni more clearly. A sample of tissue is mineralised with H_2SO_4-HNO_3, and the mixture is made alkaline with aq. NH_3 and filtered if necessary. Cryst. dimethylglyoxime (0·1 to 0·2 g) is added to an aliquot of filtrate (≡ 25 g of sample). The soln. is heated to boiling-point and set aside at room temp. for 1 hr. It is then filtered through a column (1·5 cm in diam.) containing 7 g of quartz sand (0·25 to 0·5-mm fraction). A clearly marked reddish or pink zone is formed in the upper layer. The ppt. can be removed and used for the quant. determination of Ni. E. HAYES

1596. **Determination of the chloride content of plant and animal substances by a potentiometric method.** K. Scharrer and J. Jung (Agrikulturchem. Inst., Justus Liebig Hochsch., Giessen, Germany). *Z. Tierernähr.*, 1957, **12**, 13–19.—It is not necessary to convert the substance into a dry ash. From 0·5 g to 3·0 g of finely ground air-dried material is shaken for 30 min. with 200 ml of water. From a mixture of 50 g of tungstophosphoric acid and 500 ml of conc. HNO_3, made up to 1 litre with water, a 10-ml portion is taken, added to the material and made up to 250 ml with water. After filtration, a portion is titrated electrometrically with $AgNO_3$ soln. Recovery of Cl added to plant material or to fish-meal, skimmed-milk powder or meat was satisfactory. The method is rapid and more accurate than the Volhard method. NUTR. ABSTR.

1597. **Blood alcohol calculations made easy.** P. K. van Gent (Gov. Chem. Lab., Port Elizabeth, S. Africa). *S. Afr. Med. J.*, 1957, **31**, 728–729.—The total vol. (ml) of alcohol absorbed into the body is calculated from the expression—body wt. (lb.) × blood alcohol (%) × 3·8, and the values are set out in tabular form as blood alcohol (%) of 0·05 to 0·25 and body wt. of 140 to 240 lb. The average rate of combustion of alcohol is taken as 10 ml per hr. for adults, and an alcohol elimination graph is drawn relating the time for complete elimination to the alcohol absorbed in terms of "tots" (15 ml), pints of beer (29 ml), and bottles of wine (95 ml), as well as pure alcohol (ml). Combination of the table with the graph enables all the required data to be read off without calculation.

H. F. W. KIRKPATRICK

1598. **New colorimetric method for the determination of isoniazid in biological fluids.** V. Scardi and V. Bonavita (Inst. of Human Physiol., Univ. of Naples, Italy). *Clin. Chem.*, 1957, **3** (6), 728–731 (in English).—A modified procedure, to enable the determination to be made on cerebrospinal fluid as well as serum or plasma, is proposed for the method described previously (Scardi, *Anal. Abstr.*, 1958, **5**, 184). Metaphosphoric acid (20%) (1 ml) is used to precipitate proteins in dil. serum (1 ml + 2 ml of H_2O) or fluid (2 ml + 1 ml of H_2O). To the filtrate (2 ml) is added 16·5% $(NH_4)_2HPO_4$ soln. (0·5 ml) and sodium pentacyanoamminoferroate soln. (0·5 ml), and the yellow colour is measured after 10 min. at 430 mμ, as previously described.
H. F. W. KIRKPATRICK

1599. **Concentration of chlorpromazine in human blood and its elimination in urine.** Kinjiro Iwasa, Toshio Hara, Kazuyuki Hayashi and Tatsuji Tokui (Inokashira Ment. Hosp., Tokyo). *Keio J. Med.*, 1957, **6** (2), 93–98.—The method of Dubost and Pascal (*cf. Anal. Abstr.*, 1954, **1**, 571) was used to determine chlorpromazine (**I**). The concentration of **I** in blood starts to increase 1 hr. after oral administration (600 to 700 mg), reaching a max. in 3 to 5 hr. (approx. 2 mg per litre). Excretion of **I** in the urine represents only 5 to 7% of the dose, about two-thirds being in combined form, compared with only a trace in this form in blood.
H. F. W. KIRKPATRICK

1600. **Absorbance of various protein-free filtrates of serum.** R. J. Henry and S. Berkman (Bio-Science Lab., 2231 S. Carmelina Ave., Los Angeles, Calif., U.S.A.). *Clin. Chem.*, 1957, **3** (6), 711–715.—Serum filtrates obtained with zinc hydroxide give the lowest background absorption in the range 360 to 700 mμ; those with trichloroacetic acid show the highest absorption, with tungstic acid filtrates intermediate. The possible error introduced into photometric determinations and means of correction are discussed. H. F. W. KIRKPATRICK

1601. **Test tablet [for the determination of sugar in fluids, *e.g.*, urine].** Miles Laboratories, Inc. British Patent 787,533; date appl. 8.11.55. U.S.A., date appl. 12.1.55.—The tablets contain a bismuth compound which, when treated with a reducing sugar in the presence of an alkali, gives a colour change. A suitable composition comprises bismuth subnitrate, a solid alkali (NaOH), and $CaSO_4$ and/or CaO. A drop or two of the urine specimen to be tested is placed on the surface of a tablet; with sugar-negative specimens a light-tan colour develops; with sugar-positive urine the resulting colour is greyish-black. J. M. JACOBS

1602. **Studies with a new colorimetric test for proteinuria.** A. H. Free, C. O. Rupe and I. Metzler (Miles-Ames Res. Lab., Elkhart, Ind., U.S.A.). *Clin. Chem.*, 1957, **3** (6), 716–727.—*Tablet test*—A tablet containing bromophenol blue, salicylate buffer (pH ≃3) and cellulose is placed on a clean surface. One drop of urine is placed on the tablet followed, when absorbed, by two drops of H_2O. If protein is present, a blue - green spot will remain after adding the H_2O; if negative, no material colour change is observed at the completion of the test. *Strip test*—Thick filter-paper is impregnated with a soln. containing bromophenol blue and citrate buffer (pH ≃3), dried in an oven, and cut into strips. The strips show a green to blue colour that is roughly quant. when dipped into a urine specimen containing protein, and remain yellow if protein is absent. H. F. W. KIRKPATRICK

1603. **Direct nesslerisation of blood and urine filtrates in the determination of urea (as ammonia).** J. H. Boutwell, jun. (Clin. Chem. Lab., Temple Univ., Sch. of Med., Philadelphia, Pa., U.S.A.). *Clin. Chem.*, 1957, **3** (4), 205–216.—The formation of turbidity is discussed and a new technique proposed. Diluted blood or urine is treated with a urease soln. buffered to pH 6·8 and stabilised by addition of EDTA. The proteins are pptd. with $ZnSO_4$ - $Ba(OH)_2$, and the filtrate is nesslerised with an iodinated Nessler soln. H. F. W. KIRKPATRICK

1604. **Simple turbidimetric method of estimating blood urea.** H. Lawrie (Central Path. Lab., Burnley, Lancs., England). *Clin. Chem.*, 1957, **3** (6), 691–695.—Serum, plasma or the filtrate from equal vol. of whole blood and 20% trichloroacetic acid is used. To the serum or plasma (0·2 ml) or filtrate (0·4 ml) in a clean dry tube add 50% (v/v) acetic acid (4·0 ml, or 3·8 ml for the filtrate) and a 5% soln. of xanthhydrol in methanol (0·4 ml). Mix by gentle shaking, set aside for 15 min. at room temp., and read the turbidity in an absorptiometer with a red filter. Artificial standards are unsatisfactory and a standard graph is constructed with sera of determined urea content. Addition of urea to serum may be made by adding a soln. of urea in 50% acetic acid (10 mg per ml).
H. F. W. KIRKPATRICK

1605. **Isolation and determination of urinary hydroxykynurenine.** R. R. Brown (Cancer Res. Hosp., Univ. of Wisconsin, Madison, U.S.A.). *J. Biol. Chem.*, 1957, **227** (2), 649–652.—The method is based on the increase in yellow colour caused by treating the urine with HNO_3 after purification by ion-exchange chromatography on Dowex-50 resin. The extinction is determined spectrophotometrically at 367 mμ and the amount of 3-hydroxykynurenine is ascertained from a standard graph. Kynurenine, kynurenic acid and xanthurenic acid do not interfere. J. N. ASHLEY

1606. **Detection and determination of porphobilinogen in urine.** C. Rimington, S. Krol and B. Tooth (Univ. Coll. Hosp. Med. School, London). *Scand. J. Clin. Lab. Invest.*, 1956, **8**, 251–262.—Urine contains substances, possibly thio compounds and glucuronides, that inhibit the development of a colour reaction between porphobilinogen (**I**) and a modified Ehrlich's reagent (15 g of *p*-dimethylaminobenzaldehyde in 65·6 ml of conc. HCl and 194·4 ml of glacial acetic acid). The inhibitor can be removed by adsorbing **I** on a sodium cycle Dowex-2 (50 to 100 mesh) 8% cross-linked column and washing the inhibitor out with 25 ml of H_2O. **I** is eluted with 50 ml of 0·5 *M* ammonium acetate. Recovery of **I** is 91 to 100%. CHEM. ABSTR.

1607. **The determination of ketone bodies [in blood and urine].** P. A. Mayes and W. Robson (Dept. of Biochem., King's Coll., London). *Biochem. J.*, 1957, **67** (1), 11–15.—The method described is suitable for use with small samples (0·1 ml). It depends on the conversion of the ketone bodies into acetone and conversion of this into its 2:4-dinitrophenylhydrazone, which is then dissolved in CCl_4 and determined spectrophotometrically at 350 or 410 mμ, or with a Spekker absorptiometer and an Ilford filter (600 or 601). A combined reflux and distillation apparatus is described which ensures reproducibility in the determination of β-hydroxybutyric acid, and allows the separate and direct determination of acetone plus acetoacetic acid and of β-hydroxybutyric acid from one sample. The

sample is distilled with dil. H_2SO_4 which furnishes the acetone originally present and that derived from acetoacetic acid. The residue is then oxidised with $K_2Cr_2O_7$ and H_2SO_4 and a second lot of acetone is collected and determined. With ketone levels of 40 mg per 100 ml, the standard deviation of an individual determination is $< \pm 0{\cdot}2$ mg per 100 ml.
J. N. ASHLEY

1608. **Determination of creatine in biological fluids.** D. R. Anderson, C. M. Williams, G. M. Krise and R. M. Dowben (Radiobiol. Lab., Balcones Res. Center, Austin, Texas, U.S.A.). *Biochem. J.*, 1957, **67** (2), 258–262.—Creatine is determined by means of the colour reaction with diacetyl in the presence of 1-naphthol. Inhibitors of the diacetyl reaction, arginine and creatinine are first removed by successive adsorption on columns of cellulose, Amberlite IRA-401, IRC-50 and IR-120. The creatine is eluted from the last resin with *N* Na acetate. The eluate (7 ml containing 10 to 40 μg of creatine) is mixed with 2 ml of 1·6% 1-naphthol in aq. alkali [NaOH (60 g) and Na_2CO_3 (160 g) in water (1 litre)], and 0·05% diacetyl in water (1 ml) is added. The extinction is then measured at 525 mμ after 20 min. but before 30 min. The separation and detection of creatine, creatinine and related compounds by paper chromatography is described.
J. N. ASHLEY

1609. **Application of the carbazole reaction to the estimation of glucuronic acid and glucose in some acidic polysaccharides and in urine.** J. M. Bowness (Dept. of Biochem., Univ. of Malaya, Singapore). *Biochem. J.*, 1957, **67** (2), 295–300.—A modification of the carbazole method for the determination of uronic acids is adapted for the determination of glucuronolactone, glucose and xylose in soln. containing any or all of these compounds in the concn. range of 10 to 100 μg per ml. The soln. is mixed with the carbazole - 87% H_2SO_4 reagent, and after 2·5 hr. a reading is taken with the Spekker photo-electric colorimeter, with Ilford 604 green filter. Glucuronic acid gives an intense colour at this stage. Water is then added and two further readings are taken next day, one with Ilford 605 green filter and the other with Ilford 601 violet filter. Glucose gives a more intense colour than glucuronic acid at this stage, with max. absorption in the yellow - green region. Xylose also gives a colour at this stage with max. absorption in the yellow - green region, but the colour is less intense than that given by hexoses and it has a secondary absorption max. in the violet region.
J. N. ASHLEY

1610. **The use of formaldehyde to avoid artefacts in the chromatographic determination of keto acids.** D. Cavallini and B. Mondovi (Inst. of Biochem., Univ. of Rome, Italy). *Clin. Chim. Acta*, 1957, **2** (4), 312–315 (in English).—Formaldehyde is used as a trapping agent for excess of dinitrophenylhydrazine before alkali treatment in the method previously described (*cf.* Cavallini and Frontali, *Anal. Abstr.*, 1954, **1**, 1548).
H. F. W. KIRKPATRICK

1611. **Partition chromatography of organic acids in body fluids with silica gel. Application of the method to normal human urine.** S. Meites (Dept. of Pediatrics, Ohio State Univ. Coll. of Med., Columbus, U.S.A.). *Clin. Chem.*, 1957, **3** (4), 263–269.—The extraction and chromatography of non-α-amino organic acids is described. The relative position of elution of 42 acids is tabulated. Some important acids known to be present are not eluted.
H. F. W. KIRKPATRICK

1612. **Fluorescence of ethylenediamine derivatives of adrenaline and noradrenaline.** G. F. Mangan, jun., and J. W. Mason (Walter Reed Army Inst. of Res., Washington, D.C., U.S.A.). *Science*, 1957, **126**, 562–563.—The peak emission of the noradrenaline deriv. (**I**) is at 495 mμ, and that of the adrenaline deriv. (**II**) at 525 mμ. The ratio of the fluorescence of **II** to **I** at 510 mμ is 0·71 and at 600 mμ is 4·00. The fluorescence of **I** and **II** formed in standard acetic acid is less than that observed if condensation is carried out in acetic acid which has passed through an alumina column. The average increase at 510 mμ is 9% and 30% for **I** and **II**, respectively.
H. F. W. KIRKPATRICK

1613. **Adrenaline and noradrenaline concentrations in plasma of humans and rats.** I. Gray and J. G. Young (Walter Reed Army Inst. of Res., Washington, D.C., U.S.A.). *Clin. Chem.*, 1957, **3** (4), 239–248.—Experimental evidence is presented to show that the method of Weil-Malherbe and Bone (*Brit. Abstr. C*, 1952, 306; *Lancet*, 1953, **264**, 974), with slight modifications, provides a satisfactory standard of specificity, reproducibility and recovery.
H. F. W. KIRKPATRICK

1614. **Micro-test for mucopolysaccharides by means of tolidine blue.** N. Blumenkrantz (Dept. of Ophthalmol., Hadassah Med. Sch., Jerusalem, Israel). *Clin. Chem.*, 1957, **3** (6), 696–702.—A test is described for the detection of minute quantities (0·5 μg) of hyaluronic acid and related mucopolysaccharides.
H. F. W. KIRKPATRICK

1615. **Determination of benzoic and hippuric acids by differential spectrophotometry.** H. P. Rieder (Neurol. Univ.-Poliklinik, Basle, Switzerland). *Clin. Chim. Acta*, 1957, **2** (6), 497–501.—The composition of mixtures of the acids can be read directly from the ratio of the extinctions measured at 220 mμ and 246 mμ.
H. F. W. KIRKPATRICK

1616. **Carotenoids in man. II. Fractions obtained from atherosclerotic and normal aortas, serum and depot fat by separation on alumina.** [**Determination of carotenoids.**] D. H. Blankenhorn (Metabolic Lab., Coll. of Med., Univ. of Cincinnati, Ohio, U.S.A.). *J. Biol. Chem.*, 1957, **227** (2), 963–973.—A method is described for the micro-determination of carotenoids in human serum, depot fat, and arterial wall. After hydrolysis with ethanolic KOH, the carotenoids are extracted with hexane or with a mixture of light petroleum and ethanol. They are then adsorbed on Al_2O_3 and eluted in three fractions—(*i*) with acetone - hexane (2:100), (*ii*) with acetone - hexane (20:100), and (*iii*) with ethanol - hexane (8:100). Fraction (*i*) contains β-carotene, (*ii*) contains a mixture of lycopene, esterified lutein, and an unidentified carotenoid, whilst (*iii*) consists of lutein. Each fraction is determined spectrophotometrically at 450 mμ.
J. N. ASHLEY

1617. **Determination of purines and pyrimidines by paper electrophoresis.** G. Semenza (Ist. Biochim., Univ. Genoa). *Boll. Soc. Ital. Biol. Sper.*, 1956, **32**, 1294–1298.—The electrophoretic separation of nitrogen bases cannot be performed in neutral or alkaline soln. because of the low solubility of adenine and guanine except in acid soln. At pH 3 the percentage dissociation is, for cytosine 98, adenine 92·5, and guanine 61·5. The deoxyribonucleic acid was hydrolysed, dried and dissolved in *N* HCl, 5 to 50 μg of each base in 5 μl. A horizontal electrophoresis apparatus with humid chamber was

used, with strips of Whatman No. 1 filter-paper and citrate - HCl buffer. The 5-μl samples of hydrolysate were treated with 440 V (7 to 8 mA) for 3 hr. at room temp. The strips were dried at 100° for 5 min. The spots were detected by means of the u.v. television apparatus (Tiselius *et al.*, *Arch. Biochem. Biophys.*, 1956, **65**, 132) or by Mineralight. Single spots were eluted overnight in 3 ml of 0·1 *N* HCl, or *N* HCl for guanine. The extinction values in the Beckman apparatus were 250, 260, 280 and 290 mμ. The mean values with percentage standard error for 10 samples from a single hydrolysate of deoxyribonucleic acid from calf's thymus were adenine 29·0 ± 1·67, thymine 28·5 ± 2·64, guanine 21·2 ± 3·75 and cytosine 21·3 ± 4·75, expressed as moles per 100 moles of total base recovered.

NUTR. ABSTR.

1618. **Determination of hexosamines with special reference to nervous tissue.** L. Svennerholm (Dept. Med. Biochem., Univ., Gothenburg, Sweden). *Acta Soc. Med. Upsalien.*, 1957, **61**, 287–306.—The colorimetric determination of amino sugars after their reaction with alkaline acetylacetone and *p*-dimethylaminobenzaldehyde was studied (*cf.* Elson and Morgan, *Biochem. J.*, 1933, **27**, 1824; and Blix, *Acta Chem. Scand.*, 1948, **2**, 467). By reducing the temp. to 90° during acetylation and increasing the concn. of acetylacetone, reproducibility was increased and interference by unhydrolysed mixtures of amino acids and sugars was inhibited. Absorption due to chromogens formed during hydrolysis was eliminated by either measuring the absorption at two wavelengths or by purifying the hydrolysate by cation-exchange chromatography, or by both these methods. Interfering chromogens accounted for up to 5% absorption in lipid extracts of nervous tissue, and of 10 to 15% in fat-free nervous tissue. Interference from amino acids and sugars was negligible in mucoproteins and mucopolysaccharides, but not in materials containing greater amounts of non-nitrogenous sugars. NUTR. ABSTR.

1619. **Ultra-violet spectrophotometric determination of choline.** J.-L. Delsal (Inst. Razi, Hessarek, Teheran, Iran). *Compt. Rend.*, 1957, **244** (23), 2855–2858.—From 5 to 60 μg of choline can be determined to within ±1·5% by pptn. of the chloride in pure *iso*propyl alcohol with a freshly prepared soln. of Reinecke's salt in *iso*propyl alcohol, followed by dissolution of the well-washed ppt. in methanol and measurement of the extinction of this soln. (after remaining for 30 min. in darkness) at 310 mμ (for 20 to 60 μg) or 235 mμ (for 5 to 40 μg of choline per 10 ml) in a 1-cm quartz cell, with methanol as blank. The choline reineckate should be centrifuged and washed first with a saturated soln. of the salt in *iso*propyl alcohol and then in diethyl ether. The original sample, *e.g.*, phosphatide choline, should be hydrolysed, fatty acids removed, and the choline dissolved in pure ethanol, from which soln., after centrifuging, the ethanol is evaporated *in vacuo* at <45°. Measurements at 310 mμ are less sensitive but more accurate than are those made at 235 mμ, but in all instances the sensitivity is from two to four-fold greater than when the measurements are made in acetone soln. The procedure ensures min. solubility of the choline reineckate ppt. (*cf. Brit. Abstr. C*, 1949, 236; *J. Biol. Chem.*, 1953, **205**, 803).

W. J. BAKER

1620. **Oxidation of choline by potassium dichromate. New method of determining choline.** C. Soye. *Compt. Rend.*, 1957, **245** (4), 427–429.—Optimum conditions for the oxidation of choline to betaine with $K_2Cr_2O_7$ in acid soln. are discussed. From 6 to 60 mg of choline (as hydrochloride) can be determined by this method with an error of <1%. The sample soln. is heated in a stoppered flask at 90° to 100° for 3·5 to 4 hr. with an excess of 0·1 *N* $K_2Cr_2O_7$ in 3·3 *N* H_2SO_4; after cooling quickly, the excess of $K_2Cr_2O_7$ is titrated with $Fe(NH_4)_2(SO_4)_2$, with Ba diphenylaminesulphonate as indicator. A slight modification is necessary if the oxidation is made in 3·3 *N* HNO_3. The procedure is applicable in the presence of a large amount of UO_2^{2+}.

W. J. BAKER

1621. **Colorimetric determination of 5-hydroxytryptamine in the gastro-intestinal tract of the rat and chicken treated with reserpine.** P. Correale and I. Cortese (Univ. Bari, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1956, **32**, 525–527.—The liberation of 5-hydroxytryptamine (**I**) in the gastro-intestinal tract of the rat under the influence of reserpine is slow and lasting. The value of **I** determined colorimetrically is always lower than that determined biologically. In the chicken, the decrease in the content of **I** is more pronounced; the max. difference between colorimetric and biological values is 40%. In the controls both methods give the same values. The deviation between the two methods is greatest when the amount of **I** present is least. CHEM. ABSTR.

1622. **High-voltage electrophoretic separation of serotonin (5-hydroxytryptamine) from other xanthhydrol-positive substances.** H. G. Nöller and P. Stelgens (Univ.-Kinderklinik, Heidelberg, Germany). *Clin. Chim. Acta*, 1957, **2** (4), 304–305.—Application of a voltage of 85 V per cm with 0·6 *N* formic acid - 2 *N* acetic acid as buffer soln. (pH 1·9) gives a ready separation. The 5-hydroxytryptamine moves 11 cm towards the cathode in 60 min. whilst other xanthhydrol-positive bodies remain almost stationary. This affords a simple method of determination in body fluids.

H. F. W. KIRKPATRICK

1623. **Quantitative determination of amino acids by paper chromatography.** A. A. Denisova (Central Res. Inst. of the Boot and Leather Ind., Moscow). *Biokhimiya*, 1957, **22** (4), 755–760.—Artificial mixtures of amino acids corresponding in composition to hydrolysates of collagen from ox hides are chromatographed on Whatman 3 MM paper by a one-dimensional descending technique. A mixture of *n*-butanol, acetic acid and water (4:1:1) is used as the main solvent system. Some groups of amino acids are difficult to separate (serine - glycine, glutamic acid - threonine, phenylalanine - lysine) and for these phenol saturated with buffer soln. at pH 12·0 is used. The spots are developed with 0·1% ninhydrin in water-saturated butanol. The chromatogram is photographed in transmitted light under standard conditions. Diapositives are then made from the negatives and the density of the spots is measured with a recording photometer. The area under the curves obtained is measured planimetrically. For amino acids with sharply differing R_F values, the relative error is 4 to 5%. Examples of the use of the method for determining amino acids in hydrolysates are given. Hydroxyproline cannot be determined because its spot overlaps those of histidine and glutamic acid.

E. HAYES

1624. **Paper chromatography of urinary amino acids.** J. Kolousek (Physiol. Inst. Akad. der Wissenschaften, Prague). *Naturwissenschaften*, 1957, **44** (14), 398.—Partial desalting is carried out

by cooling the urine to 5° and filtering at this temp. In other respects the usual two-dimensional chromatographic technique is followed except that Tropaeolin OOO is used as a marker for the position of the fastest running amino acids in both solvent systems. E. KAWERAU

1625. **Accuracy of quantitative paper chromatography in amino-acid determination using direct photometry.** H. R. Roberts and M. G. Kolor (Nat. Dairy Products Corp., Oakdale, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1800–1802.—Accuracy and precision data for the McFarren and Mills's maximum colour-density procedure (*Anal. Chem.*, 1952, **24**, 650) are presented. Standards of 20 common amino acids were assayed nine times by the method; eleven acids gave average deviations from the mean of $<2\%$, thirteen $<3\%$ and seventeen $<5\%$. Only lysine, *nor*leucine and tyrosine gave values $>5\%$. G. P. COOK

1626. **Rapid procedure for the separation of amino acids on paper chromatograms.** H. R. Roberts and M. G. Kolor (Nat. Dairy Products Corp., Oakdale, Long Island, N.Y., U.S.A.). *Nature*, 1957, **180**, 384–385.—The technique of horizontal solvent front development at elevated temp. enables a much more rapid separation of material than by the conventional methods of paper chromatography. The apparatus consists of a steel tank, on top of which is clamped a glass plate. A rectangular sheet of filter-paper, one end of which dips into a solvent trough, is supported inside on glass rods. The whole tank is placed in an oven for development at elevated temp. J. H. WATON

1627. **Desalting amino-acid solutions by displacement with piperidine.** D. L. Buchanan (Veterans Admin. Hosp., West Haven, Conn., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1877–1878.—A mixture of 18 amino acids and the chlorides of Na, K, Ca and Mg were placed on a column of Dowex-50 resin and were displaced with a dil. soln. of piperidine. The amino salts are eluted well before the Na^+, the first inorganic cation to emerge. G. P. COOK

1628. **Determination of amino acids as phenylthiohydantoin derivatives. I. Micro-synthesis of 3-phenyl-2-thiohydantoins from amino acids.** J. Sjöquist (Dept. Physiol. Chem., Univ. of Lund, Sweden). *Ark. Kemi*, 1957, **11** (2), 129–135.—The 3-phenyl-2-thiohydantoin derivatives of amino acids show high absorption at 269 mμ, and this is suggested as the basis of a method for determining amino acids. In the micro-synthesis described, 0·5 μmole of amino acid is converted into its hydrochloride, then dissolved in triethylamine - acetic acid buffer (pH 10·1) and treated with a twofold molar excess of phenyl *iso*thiocyanate. The resulting phenylthiocarbamoyl amino acid is converted into the 3-phenyl-2-thiohydantoin derivative by treatment with acetic acid - HCl, then dissolved in ethanol before measurement of the u.v. absorption.

II. Chromatographic separation of 3-phenyl-2-thiohydantoins of amino acids. J. Sjöquist. *Ibid.*, 1957, **11** (2), 151–160.—A procedure for the quant. determination of the thiohydantoin derivatives of the amino acids is described. The derivatives are separated by successive column and paper chromatography, and the u.v. absorption of the eluate is automatically recorded. As little as 10 μg of amino acid can be determined, and 14 acids have been separated by an appropriate system. The procedure is applied to a hydrolysate of bovine serum albumin. C. A. SLATER

1629. **Separation and detection of trifluoroacetyl amino acids.** E. C. Smith, P. M. Althouse and J. W. Shigley (Dept. of Agric. and Biol. Chem., Pennsylvania State Univ., Univ. Park, U.S.A.). *Science*, 1957, **126**, 1287–1288.—Paper electrophoresis is carried out with K H phthalate - NaOH buffer, pH 6·0, ionic strength 0·045, for 1 hr. at 600 V. The strip is dried, dipped in 1% ethanolic NaOH (1 ml of saturated NaOH soln. in 99 ml of 95% ethanol) and dried. The colour is then developed with acidic ninhydrin reagent (2% soln. of ninhydrin in acetone containing 1% of acetic acid) (Roland and Gross, *Anal. Abstr.*, 1954, **1**, 1935). By this means glycine and alanine have been separated from their trifluoroacetyl derivatives.

H. F. W. KIRKPATRICK

1630. **The ninhydrin reaction and its analytical applications.** H. Meyer (Dept. of Biol. and Colloid Chem., Hebrew Univ., Jerusalem, Israel). *Biochem. J.*, 1957, **67** (2), 333–340.—A method is described for the spectrophotometric determination of 0·5 to 6 μg of α-amino N present in amino acids or peptides. These are treated with ninhydrin under specified conditions and the extinction is measured at 408 or 578 mμ. A formula is given for the calculation of the amount of amino N. J. N. ASHLEY

1631. **Reaction of ninhydrin in acid solution with straight-chain amino acids containing two amino groups and its application to the estimation of $\alpha\epsilon$-diaminopimelic acid.** E. Work (Univ. Coll. Hosp. Med. Sch., London, England). *Biochem. J.*, 1957, **67** (3), 416–423.—At pH 1 or less, ninhydrin reacts with straight-chain $\alpha\alpha$-diaminodicarboxylic acids, including S-containing compounds, and with dibasic amino acids. The spectra of the reaction products, except that from diaminoadipic acid, all show absorption peaks at 320 to 350 mμ and sometimes also at 430 to 460 mμ. Diaminoadipic acid gives a product which resembles that from ornithine in having its max. at 515 mμ. Changes in acidity affect the shape of the absorption spectrum of the product from $\alpha\epsilon$-diaminopimelic acid only; the absorption at 440 mμ increases on decrease of the pH from 1·0 to 0·3. A method for the determination of $\alpha\epsilon$-diaminopimelic acid is based on this, provided that tryptophan, cystine, ornithine, proline and lysine are not present in excess.

J. N. ASHLEY

1632. **Proteins. XLI. Detection of the sulphur-containing amino acids and their peptides by diagonal chromatography and diagonal electrophoresis.** O. Mikeš and V. Holeyšovský (Biochem. Dept., Chem. Inst. Acad. Sci., Prague). *Chem. Listy*, 1957, **51** (8), 1497–1500.—A new chromatographic method for the detection of methionine and methionine S-dioxide has been developed. Diagonal chromatography was very suitable for the separation of a single component from the mixture. By this method, the component to be detected is displaced from the diagonal line of the other components when two-dimensional chromatography in the same solvent is used. This method was also used for two-dimensional electrophoresis with cysteine.

J. ZÝKA

1633. **Paper-chromatographic analysis of homocysteine in the presence of related thioamino acids.** E. Strack (Karl-Marx-Univ., Leipzig, Germany). *Hoppe-Seyl. Z.*, 1956, **305**, 166–169.—Homocysteine, cysteine, homocystine, cystine and methionine have been separated by means of paper chromatography. The two thiol compounds were condensed

with formaldehyde and stabilised. A suitable solvent is *n*-butanol - formic acid - water (77:10:13) yielding clearly defined spots and characteristic R_F values. CHEM. ABSTR.

1634. **Mechanism of formaldehyde incorporation into serine.** [**Determination of serine.**] R. L. Kisliuk (Dept. of Biochem., Western Reserve Univ., Cleveland, Ohio, U.S.A.). *J. Biol. Chem.*, 1957, **227** (2), 805–814.—A modification of the method of Frisell *et al.* (*Anal. Abstr.*, 1954, **1**, 1938) is described for the determination of serine; it is as sensitive as that for formaldehyde. The serine is treated with HIO_4 and the excess of reagent is removed with a Dowex-1 column, whilst the formaldehyde, arising from the serine, is quant. recovered and is determined by the acetylacetone method of Nash (*Brit. Abstr. C*, 1953, 160). J. N. ASHLEY

1635. **New paper-chromatographic method for the estimation of histidine.** K. R. Bhattacharya, J. Datta and D. K. Roy (Ind. Inst. Biochem. Exp. Med., Calcutta). *Ann. Biochem.*, 1957, **17**, 1–4.—Histidine is separated from other compounds that give the colour reaction by paper chromatography in *n*-butanol - acetic acid - water (4:1:1). The dried paper is sprayed with Na_2CO_3 soln. and then dipped in diazotised sulphanilamide. Histidine is revealed as a cherry-red spot. The paper is then dried again and the spot is cut out and eluted with 1% Na_2CO_3 soln.; histidine is determined colorimetrically in the eluate. The accuracy is within 5%. NUTR. ABSTR.

1636. **Determination of free amino-nitrogen in protein by means of thiocarbamate derivatives.** J. Baraud (Lab. de Chim. Biol., Fac. des Sci., Bordeaux, France). *Bull. Soc. Chim. France*, 1957, (7), 948–953.—An amino-acid soln. (0·01 *M*) (100 ml), buffered to a pH of 9·2 to 9·5 with borate, is warmed for 2 hr. at 50° with 5 ml of CS_2, when α-amino groups, the ε-amino group of lysine, and the imino group of proline and hydroxyproline react to give dithiocarbamates. After the removal of excess of CS_2, 5 ml of the dithiocarbamate soln. is run drop by drop, with magnetic stirring, into 50 ml of 0·01 *M* iodine brought to pH 2 by glacial acetic acid. After 20 min. the excess of iodine is determined with 0·01 *M* thiosulphate. Similar treatment of proteins enables the reactive NH_2 groups to be determined. These are provided by end-groups and ε-NH_2 of lysine only, thus enabling lysine residues to be determined in a whole protein, as the quantity of end-group is small. This method can be used to estimate 3% of lysine in 25 mg of protein. E. J. H. BIRCH

1637. **Enzymic hydrolysis of plasma proteins for microbiological assays of amino acids.** E. S. Wynne and G. T. Daye, jun. (M. D. Anderson Hosp. and Tumor Inst., Houston, Tex., U.S.A.). *Clin. Chim. Acta*, 1957, **2** (6), 502–507.—Hydrolysis with pepsin for 2 hr. at 55°, followed by trypsin for 16 hr. at 45°, yielded maximal values in microbiological assays of human plasma for arginine, glycine, histidine, *iso*leucine, tryptophan and tyrosine. H. F. W. KIRKPATRICK

1638. **Quantitative determination of serum proteins by paper electrophoresis. Rapid dyeing method.** K. M. Formusa, R. R. Benerito, W. S. Singleton and J. L. White (U.S. Dept. Agric., New Orleans, La., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1816–1819.—A quant. dyeing technique for measuring the changes in serum-protein fractions accurately and rapidly after intravenous administration of fat emulsions is described. Under standardised conditions, the dye uptake is proportional to the concn. of the protein nitrogen regardless of the serum-protein type. Direct scanning of the dyed and oiled strips by transmission densitometry gives a linear variation of integrated area with protein-nitrogen concn. over the range $\simeq$ 0·2 to 1·7 g of protein nitrogen per 100 ml with 10-μl samples. G. P. COOK

1639. **Staining of serum glycoproteins in electrophoretic strips. A useful modification in the application of the Hotchkiss - McManus method.** G. Ceriotti (Centro per lo Studio a la Cura del Tumori, Busto Arsizio, Italy). *Clin. Chim. Acta*, 1957, **2** (4), 351–352 (in English).—After electrophoresis, dry the strips at room temp. and fix for 0·5 hr. in abs. ethanol - $CHCl_3$ - acetic acid (6:3:1). Completely evaporate the fixing fluid and immerse in HIO_4 soln. (0·5%) for 10 min. Wash in several changes of H_2O, place in freshly prepared Schiff soln. for 15 min. Wash (2 to 3 min.) in 4 changes of sulphurous soln. [10% $Na_2S_2O_5$ soln. (15 ml), *N* trichloroacetic acid (15 ml), and H_2O to 200 ml], pass rapidly through 3 successive changes of acetone containing 1% of trichloroacetic acid and dry in a stream of cold air. For each 10 strips use 100 ml of Schiff soln., 400 ml of sulphurous soln., and 200 ml of acetone - trichloroacetic acid, then renew these soln. The glycoprotein bands are coloured violet on a temporarily white background. To render the background permanently white, wash (after 48 hr.) 2 or 3 times in methanol - H_2O - acetic acid (5:4:1), immerse in acetone - trichloroacetic acid and dry in a stream of cold air. H. F. W. KIRKPATRICK

1640. **The determination of hexoses in serum proteins with orcinol.** M. Schönenberger, H. Kellner, H. Südhof and H. Haupt (Behringwerk., Marburg-Lahn, Germany). *Hoppe-Seyl. Z.*, 1957, **309** (4–6), 145–157.—The orcinol test of Tillmans and Philippi (*Biochem. Z.*, 1929, **215**, 36) as modified by Sörensen and Haugard (*Ibid.*, 1933, **260**, 247) has been examined and standardised so that reproducible results can be obtained. The reagents used are orcinol (2% w/v in 20% v/v H_2SO_4) and 60% v/v H_2SO_4. The reaction mixture is heated for 30 min. at 80° and the extinction measured at 425 mμ. An equimolecular mixture of galactose and mannose is used as a standard. In the investigation of whole serum and serum proteins the method shows an error of ±2·5%. N. E.

1641. **Protein level in cerebrospinal fluid. An evaluation of some methods of determination.** S. Sethna and M. U. Tsao (Dept. of Pediatrics, Univ. of Michigan Med. Sch., Ann Arbor, U.S.A.). *Clin. Chem.*, 1957, **3** (4), 249–256.—The methods studied were (*a*) tyrosine equivalence, (*b*) sulphosalicylic acid, (*c*) trichloroacetic acid at 37°, (*d*) biuret (2 methods); the micro-Kjeldahl method was used as a standard for comparison. Results with the biuret methods were consistently high; those from the other methods showed good agreement. Similar tests with serum and plasma are described. H. F. W. KIRKPATRICK

1642. **The conversion of casein into microbial proteins in the rumen.** [**Determination of casein.**] I. W. McDonald and R. J. Hall (A.R.C. Inst. of Animal Physiol., Babraham, Cambs., England). *Biochem. J.*, 1957, **67** (3), 400–405.—A method is described for the determination of small amounts of casein in the presence of other proteins. It

depends on the removal of inorganic phosphate from the sample by dialysis, hydrolysis of the phosphate group from the casein with dil. NaOH at 37°, and determination of the inorganic phosphate so formed by a standard method. J. N. ASHLEY

1643. **Electrophoresis of human haemoglobins in starch gel.** J. A. Owen and C. Got (Dept. of Biochem., Univ. of Melbourne, Victoria, Australia). *Clin. Chim. Acta*, 1957, **2** (6), 588–590.—The electrophoretic method is essentially that of Smithies (*Biochem. J.*, 1955, **61**, 629). The gel is prepared from sol. starch hydrolysed in HCl - acetone (1:100, v/v) at 38·5° for 75 min. and contains 14 g of starch in 100 ml of 0·0275 *M* borate buffer (pH 8·4). Haemoglobin soln. is prepared by haemolysing washed cells with water and diluting to a concn. of approx. 2 g per 100 ml. Electrophoresis is carried out at 20° for 3 hr. under a potential gradient of 12 V per cm. By this technique it is possible to separate haemoglobin A from haemoglobin F.
H. F. W. KIRKPATRICK

1644. **Chromatographic behaviour of different abnormal human haemoglobins on the cation exchanger Amberlite IRC-50.** T. H. J. Huisman and H. K. Prins (Dept. of Paedriat., State Univ. of Groningen, The Netherlands). *Clin. Chim. Acta*, 1957, **2** (4), 307–311 (in English).—In this extension of previous work (*cf. Anal. Abstr.*, 1955, **2**, 2498; 1956, **3**, 172), a description is given of the chromatography of eight different types in addition to the normal adult haemoglobin.
H. F. W. KIRKPATRICK

1645. **The use of diacetin in pre-staining serum lipoproteins.** P. Solinas, R. Betti and E. F. P. Di Leo (Inst. of Gen. Clin. Med. and Therap. Med., Univ. of Perugia, Italy). *Clin. Chim. Acta*, 1957, **2** (6), 586–587 (in English).—A simple modified pre-staining procedure is proposed. To blood serum (1 ml) add a saturated diacetin soln. of acetylated Sudan black B (0·1 ml), slowly and with gentle shaking. Set aside at room temp. for 30 min., and centrifuge to eliminate any pptd. dye. The α- and β-lipoproteins in this pre-stained serum appear as blue zones on a white background after electrophoresis, and the strips may be scanned directly after drying. H. F. W. KIRKPATRICK

1646. **Determining serum protein-bound iodine.** R. D. Strickland and C. M. Maloney (Veterans Admin. Hosp., Albuquerque, N.M., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1870–1873.—The method for determining iodine in serum by its catalytic effect on the reaction between cerate and arsenite was investigated to determine the optimum concn. of Cl^- and acid that affect the rate of this reaction. The method was improved by (*a*) addition of Hg^{2+} to stabilise the cerate after incubation and (*b*) titrimetric determination of the cerate with ferrous 2:2′-dipyridyl. The standard deviation at the 5 μg per 100 ml level was ±0·224 and at the 10 μg per 100 ml level it was ±0·369 from 12 replicate standard samples. The range of the concn. of protein-bound iodine in normal subjects is estimated at 3·60 to 7·36 μg per 100 ml. G. P. COOK

1647. **Chromatographic separation of some steroids by phase inversion.** S. Bruno (Univ. Bari, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1956, **32**, 530–532.—Steroids were separated by the use of paper impregnated with chromic chloride stearate or tungstosilicic acid as the stationary phase and methanol or ethanol as the solvent. CHEM. ABSTR.

1648. **Quantitative determination of cholesterol in serum with toluene-*p*-sulphonic acid.** T. J. Turner and L. Eales (Groote Schuur Hosp., Cape Town). *Scand. J. Clin. Lab. Invest.*, 1957, **9**, 210 (in English).—Serum (0·2 ml) is mixed with 0·5 ml of glacial acetic acid and 5·0 ml of acetic anhydride in a stoppered centrifuge tube. After 5 to 15 min. the mixture is centrifuged at 2000 r.p.m. for 10 min. Then 3·0 ml of the supernatant fluid is transferred to a colorimeter tube, 1·0 ml of toluene-*p*-sulphonic acid soln. (12% in glacial acetic acid) is added and, 30 min. later, 0·4 ml of conc. H_2SO_4. After another 20 min. the colour is read at 550 mμ. For the blank, serum is replaced by H_2O. For the standard, serum is replaced by 0·2 ml of H_2O and the glacial acetic acid by 0·5 ml of a soln. of 80 mg of cholesterol in 100 ml of glacial acetic acid. NUTR. ABSTR.

1649. **Estimation of total cholesterol in serum by a micro-method.** K. J. Carpenter, A. Gotsis and D. M. Hegsted (Harvard Sch. of Public Health, Boston, Mass., U.S.A.). *Clin. Chem.*, 1957, **3** (4), 233–238.—Measurement of the fluorescence after addition of H_2SO_4 to a soln. of cholesterol in a mixture of 1:1:2-trichloroethane and acetic anhydride (*cf.* Albers and Lowry, *Anal. Chem.*, 1955, **27**, 1829) is applied to the determination in 0·02 ml of blood serum. H. F. W. KIRKPATRICK

1650. **Micro-determination of cholesterol by use of 0·04 ml of blood serum.** L. S. Galloway, P. W. Nielson, E. G. Wilcox and E. M. Lantz (Utah Expt. Sta., Logan, U.S.A.). *Clin. Chem.*, 1957, **3** (4), 226–232.—The method of Sperry and Webb (*Brit. Abstr. C*, 1951, 219) is adapted for use with micro techniques. H. F. W. KIRKPATRICK

1651. **Determination of carbon-14 steroids on paper chromatograms.** D. L. Berliner, O. V. Dominguez and G. Westenskow (Univ. of Utah Coll. of Med., Salt Lake City, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1797–1800.—The major points of loss in the chromatographic determination of radioactive steroids are the stages involving elution and plating. Steroids may be determined directly on the paper by using β-ray self-absorption factors and by measuring radioactive areas on a strip counter. The coefficients of variation were generally $< \pm 15\%$ over the range 240 to 40,000 counts per minute of directly applied steroid.
G. P. COOK

1652. **Colorimetric determination of pregnanediol in urine.** A. Martin and L. S. de Caligaris (Inst. Med. Claudio Bernard, Avellaneda 280, Cordoba, Argentina). *Rev. Soc. Argent. Biol.*, 1957, **33** (1–2), 27–35.—The method includes purification steps to overcome lack of specificity of the H_2SO_4 reaction. Urine (100 ml) is boiled for some minutes with 50 ml of toluene and 15 ml of conc. HCl, then cooled, and the extract is washed twice with 20 ml of *N* NaOH, followed by gentle refluxing, with the addition every 5 min. of a KOH pellet until the product is almost colourless. The solvent is removed and the residue extracted fractionally with 5 ml of acetone; the extract is then boiled, with slow addition of 20 ml of 0·1 *N* NaOH. After 3 min. it is cooled and refrigerated for 1 hr., when pregnanediol is pptd. The ppt. is washed twice with 10 ml of water, dissolved fractionally in 10 ml of abs. ethanol, and made up to 50 ml with water, then refrigerated for 1 day. The ppt. is washed and re-dissolved and the soln. evaporated to dryness. The residue is treated with 10 ml of conc.

H_2SO_4 for 1 hr. and the colour is measured at 430 mμ against an acid blank. The method gives a recovery of 76% starting from the Na glucuronidate; the m.p. varies from 205° to 228°.
G. F. PENNY

1653. **A test for qualitative and quantitative estimation of pregnane-3α:17α:20α-triol-11-one in urine, and its significance in adrenal disturbances.** M. Finkelstein and S. Goldberg (Hebrew Univ., Hadassah Med. Sch., Jerusalem, Israel). *J. Clin. Endocrin. & Metabolism*, 1957, **17** (9), 1063–1070.—Qual. detection of the steroid is made, after paper chromatography of urine extracts, by dipping the paper briefly in 70% H_3PO_4, heating at 87° for 10 min. and examining for a blue fluorescence in u.v. light. The amount is determined quant. by eluting the corresponding area of an untreated chromatogram and measuring the fluorescence produced by heating for 20 min. at 100° with 85% H_3PO_4. The steroid is present in the urine of patients with adrenal hyperplasia, but cannot be detected in the urine of normal subjects or of patients with adrenal tumour.
H. F. W. KIRKPATRICK

1654. **Use of phytic acid for the determination of peptic activity in gastric juice.** J. E. Courtois and H. Villiers-Huiban (Lab. de Chim. Biol., Fac. de Pharm., Paris, France). *Clin. Chim. Acta*, 1957, **2** (4), 332–339.—The gastric juice is incubated at 37° for 1 hr. with a substrate of edestin buffered to about pH 2·2. Undigested protein is then pptd. with an acid (HCl) soln. of phytic acid, separated, and determined by the biuret reaction.
H. F. W. KIRKPATRICK

1655. **Analysis for thrombin and the inactivation of fibrin monomer.** S. Ehrenpreis and H. A. Scheraga (Dept. of Chem., Cornell Univ., Ithaca, N.Y., U.S.A.). *J. Biol. Chem.*, 1957, **227** (2), 1043–1061.—The variables in the toluene-*p*-sulphonyl-L-arginine methyl ester assay method for thrombin are investigated, and an accurate and rapid method is described for the determination of the enzyme. The modifications to the literature method (Sherry and Troll, *J. Biol. Chem.*, 1954, **208**, 95) include hydrolysis of the substrate by the enzyme at 24° to 26° and titration of the acid liberated with 0·05 *N* NaOH, the temp. being kept constant.
J. N. ASHLEY

1656. **Methods for the determination of the glucokinase reaction in liver.** R. M. Bekina and A. N. Petrova (Lab. Physiol. Chem., Acad. Sci. USSR, Moscow). *Biokhimiya*, 1957, **22** (4), 636–643.—Two methods are compared—(*i*) based on the decrease in glucose and (*ii*) based on the decrease in adenosine triphosphate. The first method is unreliable but the second gives satisfactory results. Factors affecting the reaction are examined, and details are given of a procedure for treating liver samples that reduces interference from other reactions to a minimum.
E. HAYES

1657. **Spectrophotometric methods for the determination of cholinesterase activity.** E. M. Gal and E. Roth (Vet. Admin. Hosp., Sepulveda, Calif., U.S.A.). *Clin. Chim. Acta*, 1957, **2** (4), 316–326.—Two methods are described in which thiocholine esters are used as substrates. In the first, the enzyme activity is measured by the rate of disappearance of the C–S bond at 229 mμ; in the second, the reduction of indophenol by the liberated thiocholine is measured in the visible spectrum at 600 mμ. Micro amounts of blood, serum or tissue extracts are used.
H. F. W. KIRKPATRICK

1658. **Semi-micro technique for the measurement of NN-dimethyl-*p*-phenylenediamine (DPP) oxidase in blood.** L. Goldschmidt (Creedmoor Inst. for Psychobiological Studies, Queens Village, N.Y., U.S.A.). *Clin. Chem.*, 1957, **3** (6), 738–743.—The proposed method requires only a small amount of sample (0·2 ml), and enables rapid and reproducible serial determinations to be made. The oxidase in the blood remains stable for several days.
H. F. W. KIRKPATRICK

1659. **Improved method for the colorimetric determination of glutamic-oxalacetic transaminase activity.** F. L. Humoller, J. M. Holthaus and J. R. Walsh (Vet. Admin. Hosp., Omaha, Nebr., U.S.A.). *Clin. Chem.*, 1957, **3** (6), 703–710.—The proposed method is a modification of that of Cabaud *et al.* (*Anal. Abstr.*, 1957, **4**, 2352). The pyruvate dinitrophenylhydrazone is extracted with an aq. soln. of $NaHCO_3$ to eliminate the interference of excess of dinitrophenylhydrazine.
H. F. W. KIRKPATRICK

1660. **Collagen. I. Partial purification, assay, and mode of activation of bacterial collagenase.** P. M. Gallop, S. Seifter and E. Meilman (Dept. of Med. and Lab., Long Island Jewish Hosp., N.Y., U.S.A.). *J. Biol. Chem.*, 1957, **227** (2), 891–906.—Three separate methods are described for the assay of collagenase, with isinglass as substrate. In two of them the substrate is used as a suspension and in the other a soln. of the substrate in 0·5 *M* $CaCl_2$ at pH 7·0 is used. In the first two methods the extent of enzymic activity is determined by measuring the amount of collagen which passes into soln., and in the third method the activity is measured by determination of the change of viscosity of the soln. with time. This method is very sensitive.
J. N. ASHLEY

See also Abstracts—**1499**, Determination of traces of selenium in biological matter. **1513**, Microdetermination of fluorine. **1661**, Determination of ethanol by Widmark's method.

Drugs

1661. **Report of the Conference on "Some Questions of Pharmacy."** Kiev, Gosmedizdat, UkrSSR, 1956.—The following papers read at the Conference have been abstracted in *Ref. Zhur., Khim.*, 1957, (13). The page numbers of the report and the abstract numbers are given after each title. **New reactions for sulphanilamide preparations,** R. M. Zaĭtseva, 23–28; 44,946. **Quantitative determination of procaine and amethocaine by using thiocyanate complexes,** A. I. Portnov and R. M. Zaĭtseva, 29–30; 44,942. **New qualitative reaction for penicillin,** A. I. Portnov and S. G. Pligin, 31–32; 44,947. **New method for the quantitative determination of penicillin,** S. G. Pligin, 33–41; 44,948. **Quantitative determination of alkaloids as thiocyanate complexes,** V. I. Crlova and A. I. Portnov, 42–47; 44,936. **Micro-crystalloscopic reactions for nicotinic acid, nikethamide, mepacrine and procaine,** P. O. Knizhko, A. I. Zakin and V. A. Grizo, 66–68; 44,941. **The use of chloropalladic acid in microchemical analysis of organic substances,** P. O. Knizhko, A. I. Zakin and V. A. Grizo, 69–71; 44,944. **Quantitative determination of riboflavine,**

V. F. Kramarenko and A. P. Levtova, 86–88; 44,951. **Chronometric micro-analysis,** V. G. Gurevich, E. M. Skibina, N. E. Khomenko and I. V. Emel'yanenko, 89–93; 44,802. **Use of chromatographic and luminescence analysis in the determination of the alkaloids of the tropane group (atropine and hyoscine),** E. S. Binova, 113–117; 44,937. **Quantitative determination of lead with 4-aminosalicylic acid,** A. I. Vasyutinskiĭ, 133–136; 44,859. **Fluorimetric determination of folic acid,** I. R. Gnidets, 137–139; 44,924. **Use of the micro-method in the determination of ethanol by Widmark's method in chemico-legal research,** E. S. Baranova and T. V. Marchenko, 140–144; 44,916. **Analysis of rhodanine and its derivatives,** E. V. Vladzimirskaya, 145–146; 44,943. **Characteristic reactions for Tibione (thiacetazone) and Phthivazid (acetaldehyde *iso*nicotinoylhydrazone),** V. F. Gevlich, 147–149; 44,945. **Ammoniacal solution of picric acid for the micro-crystalloscopic analysis of a group of alkaloids,** V. T. Pozdnyakova, 150–154; 44,935.

C. D. KOPKIN

1662. **A micro-quantitative determination of alkaloids with Tropaeolin OO.** A. Haussler (Pharm. Lab. Farbwerk Hoechst A.-G., Frankfurt a.-M., Germany). *Dtsch. ApothZtg*, 1957, **97** (33), 729–731.—The colorimetric reaction of Tropaeolin OO with alkaloids under acid conditions is used as a micro quant. method. The aq. soln. of the alkaloid (5 ml containing $\simeq$ 100 μg of alkaloid) is mixed with a similar quantity of an acetate buffer of pH 4·6, and 3 ml of a satd. aq. Tropaeolin OO soln. is added. This soln. is then extracted with chloroform (4 × 5 ml), the combined extracts are acidified with 2 ml of an acid reagent (1 ml of conc. H_2SO_4 and 99 ml of methanol), and diluted to 25 ml with chloroform. The alkaloid is then determined spectrophotometrically at 545 mμ, and the results are compared with standard curves.

G. R. WHALLEY

1663. **Electrochromatophoretic isolation of alkaloids.** C. L. Brown and P. L. Kirk (School of Criminology, Univ. of Calif., Berkeley, U.S.A.). *Mikrochim. Acta*, 1957, (5), 720–723 (in English).—Small amounts of strychnine, morphine and quinine can be separated from impure samples and biological materials by a process of continuous electrochromatophoresis (*cf. Anal. Abstr.*, 1957, **4**, 1066). The purity of the products is sufficient for the application of the common crystal tests. Alternatively, the alkaloids are pptd. as picrates on the paper by allowing picric acid to migrate across the paper in the opposite direction to the alkaloids.

A. R. ROGERS

1664. **The quantitative oscillographic polarography of certain alkaloids.** L. Molnár (Inst. of Chem., Slovak Acad. of Sci., Bratislava, Czechoslovakia). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 273–278.—Veratrum and quinine alkaloids give characteristic oscillograms which can be used for their determination with an accuracy of $\pm 4\%$.

G. H. FOXLEY

1665. **Preliminary report on the use of ammonium reineckate in the determination of opium alkaloids.** L. Kum-Tatt and C. G. Farmilo (Dept. Chem., Outram Road, Singapore). U.N. Secretariat Publn. ST/SOA/SER.K/49, 1957, 11 pp.—Pure alkaloids, or the alkaloids in the isolates obtained in a unified analysis of opium, may be estimated by quantitative pptn. by ammonium reineckate followed by spectrophotometric evaluation of the soln. of the ppt. in acetone. *Procedure*—Dissolve 5 to 10 mg of the alkaloid or one of its salts in *N* HCl (5 ml), cool in an ice-bath and add freshly prepared ammonium reineckate reagent [triturate ammonium reineckate (2·0 g) with ice-cold H_2O (2 × 50 ml) and filter through a No. 42 Whatman paper]. Allow the beaker to stand in the ice-bath until its contents are at 0°. Collect the ppt. in a sintered glass funnel and wash with ice-cold H_2O (1-ml portions) until the wash liquor is colourless. Dry the ppt. with suction, and transfer it to a 10-ml flask with acetone. Read the extinction of the red soln. at 524 mμ, then $W/M = (E/106{\cdot}5)10$, where W = mg of alkaloid or salt taken and M = mol. wt. of alkaloid or salt and E is the extinction. An efficient method for the separation of morphine and codeine by the use of an anion-exchange resin (IRA-400) is described.

H. B. HEATH

1666. **The unified analysis of opium by paper chromatography and spectrophotometry.** Haruyo Asahina and Masako Ono (Nat. Hyg. Lab., Tokyo, Japan). U.N. Secretariat, Publn. ST/SOA/SER.K/50, 1957, 9 pp.—A micro-analysis of opium by which the five main alkaloids can be assayed from a sample of 0·1 g or less is described. *Procedure for morphine*—Accurately weigh approx. 0·1 g of air-dry opium, add 0·1 *N* HCl (1 ml) and mix to form a homogeneous paste, set aside for 60 min. then centrifuge for 5 min. Spot 0·003 ml of the supernatant soln. for chromatography, develop with the top layer of a mixture of *n*-butanol, aq. NH_3 (24%) and H_2O (50:9:15) and read the extinction of the morphine spot at 286 mμ. *Codeine*—Extract 0·5 ml of the acid soln. of opium with 2 × 2 ml and 3 × 1 ml of $CHCl_3$. Spot 0·005 ml of the aqueous phase for chromatography, develop as for morphine, and read its optical density at 283 mμ. *Thebaine-papaverine*—Combine the $CHCl_3$ extracts and evaporate on a water bath. Dissolve the residue in benzene (3 ml) and alcoholic KOH (10% w/v) (3 ml), set aside for 40 min. then transfer to a separating funnel with the aid of benzene (3 × 3 ml) and *N* NaOH (6 ml). Shake, separate and wash the benzene layer with *N* NaOH (2 × 3 ml), followed by H_2O (2 × 3 ml). Filter the benzene layer, evaporate and dissolve the residue in 0·1 *N* HCl (0·3 ml). Spot 0·006 ml of this soln. for chromatography, develop with a H_2O-satd. mixture of *n*-butanol (100 ml) and acetic acid (4 ml), and read the extinction of the thebaine spot at 285 mμ and the papaverine at 281 mμ. *Narcotine*—Combine the aq. soln. and filter; add HCl (3 ml) and heat at 95° for 30 min.; cool and extract with 2 × 10 ml and 2 × 5 ml of $CHCl_3$. Evaporate the $CHCl_3$ soln. and dissolve the residue in 0·1 *N* HCl (0·3 ml). Spot 0·003 ml of this soln. for chromatography, develop with the upper layer of a mixture of *n*-butanol, acetic acid and H_2O (5:1:4) and read the extinction of the spot at 290 mμ. Run standards at two levels for each alkaloid and from the standard curve calculate the alkaloid content in the opium.

H. B. HEATH

1667. **The use of ion exchangers in the chemistry of alkaloids. III. Isolation of morphine from opium.** J. Böswart and A. Jindra (Res. Inst. Medicinal Plants, Prague). *Československ. Farm.*, 1957, **6** (3), 145–147.—The applicability of ion exchangers depends on the method of extraction of morphine from opium. In general, the largest amount of morphine is obtained by extraction with a satd. soln. of $Ba(OH)_2$. For anion exchangers, hot-water extracts are most suitable; for cation exchangers either hot water or $Ba(OH)_2$ soln., which is neutralised after-

wards. The final analyses were performed polarographically. From the results it was concluded that Lewatit MN and Wofatit F are suitable exchangers.
J. VOLKE

1668. **Use of EDTA (disodium salt) in the determination of alkaloids. I. Determination of quinine hydrochloride.** V. A. Zaĭtsev (Moscow Pharm. Inst.). *Aptechnoe Delo*, 1957, **6** (4), 48–51.—Quinine is pptd. by CdI_2 in KI soln., and the excess of Cd is determined complexometrically. *Procedure*—Dissolve a weighed sample (0·02 to 0·05 g) of quinine hydrochloride in 10 ml of water. Add 10 ml of CdI_2 in KI soln. (dissolve 20 g of KI and 1 g of CdI_2 in 80 ml of water) and make up to 50 ml. Set aside for 15 min. and then filter through a dry filter. Reject the first 10 to 15 ml of filtrate; to a 20-ml quantity of the remainder add 30 to 40 ml of water and heat to boiling. Add 15 ml of buffer soln. (20 g of NH_4Cl and 100 ml of conc. aq. NH_3 made up to 1 litre) and "acid chrome dark blue" indicator (0·08 to 0·1 g). Titrate with 0·05 *N* EDTA (disodium salt) until a blue-violet colour is produced. The sharpest end-point is obtained at 50° to 60°.
E. HAYES

1669. **Quantitative determination of some salts of alkaloids and anaesthetics in ampoule solutions with the aid of "anionite H-O."** D. S. Yaskina (Moscow Pharm. Inst.). *Aptechnoe Delo*, 1957, **6** (4), 46–48.—A soln. of atropine hydrochloride, hyoscine hydrobromide or procaine hydrochloride is passed through a column 12 mm in diam. containing 10 g of "anionite H-O" (0·8 to 2·0-mμ fraction) (characteristics given). The column is washed with ethanol and water and the base is determined acidimetrically in the eluate.
E. HAYES

1670. **Colorimetric determination of organic base preparations with sulphonephthalein dyes. I. Light absorption and partition coefficient of various sulphonephthalein dyes.** Masayoshi Horioka (Hosp. Pharm., Tokyo Univ., Hongo). *J. Pharm. Soc. Japan*, 1957, **77** (2), 200–206.—The extraction of addition compounds of sulphonephthalein dyes and organic bases, *e.g.*, methylephedrine hydrochloride (**I**) and ephedrine hydrochloride (**II**), with 1:2-dichloroethane (**III**), $CHCl_3$, benzene, CCl_4 or butyl acetate, is best achieved at pH 5 to 6; at a lower pH the dye itself is extracted. When the dye is extracted back with 0·1 *N* NaOH, the extinction due to the dye is proportional to the amount of the organic base present in the original soln., provided that the procedure is carried out in accordance with a given prescription. Dyes containing halogens, *e.g.*, bromothymol blue, bromocresol green (**IV**) and bromocresol purple, are suitable for the analysis.

II. Determination of sympathomimetic amines in pharmaceutical preparations. Masayoshi Horioka. *Ibid.*, 1957, **77** (2), 206–209.—Preparations of **I**, **II**, amphetamine sulphate, methoxyphenamine, etc., are mixed with **IV** in Sørensen's buffer (pH 5·60) and extracted with **III**. The dye is extracted back with 0·1 *N* NaOH and the extinction measured at 617 mμ. No interference results from other ingredients in such preparations (*e.g.*, saccharides, NaCl, antipyretics, sedatives). Antihistamines such as diphenhydramine and alkaloids cause interference. The working curve is linear for <50 μg of **I** and <100 μg of **II** per ml of the original soln. *Procedure*—Mix aq. soln. of the sample (2 ml) (centrifuge, when necessary) with the buffer (4 ml) and 0·1% **IV** (2 ml), shake with **III** (20 ml) for 10 min. and centrifuge. Shake the **III** layer (10 ml) with 0·1 *N* NaOH (10 ml) for 2 min. and measure the extinction of the aq. layer.
K. SAITO

1671. **Applications of the amperometric titration method in the analysis of pharmaceutical preparations.** G. Bozsai (Dept. for Chem., State Inst. for Public Health, Budapest). *Acta Chim. Acad. Sci. Hung.*, 1956, **9** (1–4), 265–272.—Tervalent As is oxidised to the quinquevalent form by $K_2Cr_2O_7$, and the excess of the reagent is determined by amperometric titration with Fe^{2+}. Codeine hydrochloride in the presence of other drugs, *e.g.*, quinine sulphate and acetylsalicylic acid, can be determined by amperometric titration with Ag^+ by using a rotating platinum electrode and a calomel electrode. Other examples given are the detection and determination of ZnO in various preparations by titration with $K_4Fe(CN)_6$ soln., and the oxidation of phenetidine by ceric salts.
G. H. FOXLEY

1672. **Application of photocolorimetry to the analysis of some pharmaceutical mixtures.** L. I. Rapaport (Central Res. Lab. "GAPU," Min. of Health UkrSSR). *Aptechnoe Delo*, 1957, **6** (3), 21–28.—Methods are described for the determination of phenacetin, "izafenin" (diacetyldiphenyloxindole) and strychnine in pharmaceutical mixtures. For the determination of phenacetin, a weighed sample (0·02 g) is dissolved in boiling H_2O (15 ml) and 12 ml of dil. HNO_3 (sp. gr. 1·095) is added; the mixture is set aside for 2 min., diluted with hot water, cooled and made up to 250 ml. The yellow colour is then measured. Acetanilide, caffeine, quinine, Na benzoate, Na salicylate, acetylsalicylic acid, phenobarbitone, barbitone, hexamine, starch, talc and codeine do not materially interfere, but phenazone, phenazone salicylate and amidopyrine do. Izafenin is hydrolysed with NaOH, and $K_3Fe(CN)_6$ is added to the acidified soln. The colour produced is measured in an absorptiometer fitted with a dark-blue filter. Sucrose, talc and starch do not interfere. The determination of strychnine is based on the reaction of its reduction products with $NaNO_2$ to give a pink colour.
E. HAYES

1673. **Determination of khellin in the fruits of *Ammi visnaga* by paper chromatography.** Toyohiko Kawatani, Tadaro Ohno and Miyoko Ito. *Eisei Shikenjo Hôkoku*, 1956, (74), 73–76.—Suspend the minced fruit in water (0·5 g in 100 ml), boil, filter, and precipitate impurities with Pb acetate and Na_2HPO_4 in succession. Extract the khellin (**I**) with $CHCl_3$ (4 × 25 ml), dehydrate the soln. with Na_2SO_4, distil off the $CHCl_3$, and dissolve the syrupy residue in ethanol. Chromatograph the soln. containing 50 to 300 μg of **I** on paper with acetic acid - water (6:4) for 15 hr., and detect the fluorescent spot of **I** ($R_F = 0·79$). Extract **I** from the spot with methanol, evaporate, add 10 *N* H_2SO_4 to the residue, and determine the extinction at 410 mμ.
CHEM. ABSTR.

1674. **Characterisation and purification of subtilin by paper electrophoresis.** L. E. Sacks and J. W. Pence (West Reg. Res. Lab., Albany, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1802–1805.—The occurrence of multiple polypeptides in subtilin preparations was demonstrated by the technique of hanging-curtain paper electrophoresis coupled with peptide staining. Of the two chief components isolated by this method, the major one, termed subtilin *A*, possessed high anti-bacterial activity against *Arthrobacter citreus*; the other, subtilin *B*, had low anti-bacterial activity.
G. P. COOK

1675. **Polarographic determination of bromoform in drugs.** V. Volková and F. Icha (Polarographic Inst. Acad. Sci., Prague). *Českosl. Farm.*, 1957, **6** (3), 141–145.—Sodium bromide (0·1 *M*) is recommended as supporting electrolyte with gelatin as maximum suppressor. Because of the volatility of bromoform, oxygen is removed from the soln. with sodium sulphite. Various extracts, tinctures, alkaloids, halogen salts, and up to 33% of sucrose in the analysed soln. do not interfere. The analytical procedure and the probable mechanism of reduction are described. J. Volke

1676. **Quantitative determination of methyl salicylate in pharmaceutical mixtures.** G. A. Vaĭsman and S. N. Benderskaya (Control Lab. of Kiev District Pharm. Admin.). *Aptechnoe Delo*, 1957, **6** (2), 32–36.—The sample is saponified with aq. alkali, the soap is pptd. with $BaCl_2$, and the Na salicylate is determined bromimetrically. *Procedure*—A weighed sample containing 0·3 to 0·4 g of methyl salicylate is heated under reflux in a 100-ml conical flask with 20 ml of *N* NaOH for 10 min., with frequent shaking. The cooled liquid is filtered through moistened filter-paper, and the filtrate is made up to 100 ml. To a 5 to 10-ml aliquot of the filtrate is added 5 ml of a 5% soln. of $BaCl_2$, and the mixture is heated on a water bath for 15 min. The cooled soln. is filtered into a 100 to 200-ml stoppered flask; 1 g of KBr, 25 ml of 0·1 *N* $KBrO_3$ and 5 ml of dil. HCl are added, and the flask is set aside in the dark for 15 min.; 1 g of KI is added and the liberated iodine is titrated with $Na_2S_2O_3$. The method has been applied successfully to several complex pharmaceutical mixtures. E. Hayes

1677. **Ion exchangers in pharmaceutical analysis. X. Studies on the behaviour of barbiturates.** A. Jindra and F. Balák (Pharm. Control Inst., Prague). *Českosl. Farm.*, 1957, **6** (3), 148–150.—Barbiturates are quant. adsorbed on the strongly basic ion exchangers Amberlite 1RA-400 and Lewatit MN from ethanolic soln. and eluted with 0·2 *M* acetic acid. The following compounds were investigated—barbitone, phenobarbitone, allobarbitone, aprobarbital, narcobarbital and thiopentone. The contents in the eluate were determined gravimetrically, spectrophotometrically and by alkalimetric and bromatometric titrations. J. Volke

1678. **New volumetric methods for the analysis of organic compounds. IV. Utilisation of addition reactions of N-bromosuccinimide.** A. Berka and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Českosl. Farm.*, 1957, **6** (4), 212–215.—Several compounds containing an allyl group were titrated potentiometrically with 0·01 *M* N-bromosuccinimide in HCl (in some cases, with methyl red as indicator). The bromination of aromatic compounds with the same reagent proceeds slowly. The standardisation of the reagent is described. Allyl*iso*butyl-, allyl*iso*propyl- and diallyl-barbituric acids, allyl alcohol, allylamine, crotonic acid and amides of tetrahydrobenzoic acid may be determined with an error usually $< \pm 1\%$. Barbiturates with a benzene nucleus interfere. J. Volke

1679. **Analysis of aminophenazone in the control of amidopyrine production.** L. M. Litvinenko, V. P. Polyakov, A. P. Grekov and A. M. Chernetskaya. *Med. Prom., SSSR*, 1957, (1), 46–48; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,784.—Instead of the previously used diazometric titration for the analysis of aromatic amines, potentiometric titration with $NaNO_2$ soln. is recommended; impurities are first pptd. with Pb acetate. C. D. Kopkin

1680. **Determination of the composition of binary pharmaceutical mixtures by fusion temperatures.** N. A. Figurovskiĭ and V. M. Bashilova (Central Pharm. Research Inst. Min. of Health, USSR). *Aptechnoe Delo*, 1957, **6** (2), 5–10.—Fusion diagrams are given for (*i*) amidopyrine - acetylsalicylic acid, (*ii*) phenacetin - acetylsalicylic acid, (*iii*) amidopyrine - phenacetin, (*iv*) amidopyrine - caffeine, (*v*) phenobarbitone - amidopyrine, and (*vi*) phenobarbitone - phenacetin. For these mixtures, the proportion of each constituent present can be determined from the m.p.; when the m.p. indicates two possible mixtures, the composition is found by adding a known amount of one component and redetermining the m.p. Amidopyrine - phenazone does not yield a sharp eutectic. E. Hayes

1681. **Qualitative reactions for pethidine, methadone and trimeperidine (1:2:5-trimethyl-4-phenyl-4-propionyloxypiperidine hydrochloride).** R. M. Ternikova (Moscow Pharm. Inst.). *Aptechnoe Delo*, 1957, **6** (2), 38–43.—The behaviour of pethidine, methadone and trimeperidine with 11 alkaloidal reagents is examined and the results obtained are tabulated; sensitivities are also reported. A table showing how the compounds react with the following reagents is given—(*i*) H_2SO_4 (sp. gr. 1·84), (*ii*) conc. H_2SO_4 - molybdic acid, (*iii*) conc. H_2SO_4 - conc. HNO_3, (*iv*) conc. H_2SO_4 - vanadic acid, (*v*) conc. H_2SO_4 - formaldehyde. The most useful reagent is (*v*); with pethidine this gives a slight pink colour rapidly turning brown; with methadone it gives a black colour on heating; and with trimeperidine a reddish-purple colour turning to brown. E. Hayes

1682. **Extraction and characterisation of sympathomimetic amines.** A. Hasson (Inst. Biofísica, Brazil Univ., Rio de Janeiro). *An. Acad. Brasil. Cienc.*, 1957, **29** (1), 27–37.—Quant. recovery of monophenolic amines is carried out by adsorption on activated charcoal, and semi-quant. recovery by solvent extraction. The extracts are analysed by electrophoresis and chromatography on filter-paper. Diazotisation reactions are used for the identification of 5 μg of phenolic amines, and 10 μg of non-phenolic amines can be identified by complex formation. R_F values are given for the hydrochlorides of ephedrine, amphetamine, dexamphetamine, methylamphetamine, hydroxyamphetamine, phenylephrine, 2-aminoheptane, adrenaline and noradrenaline with various solvent systems; mobilities are given for the same group in various electrolytes; and colour reactions for the same group except dexamphetamine. G. F. Penny

1683. **Determination of some new antihistamines by a chromatographic ion-exchange method.** J. Blažek, V. Mareš and Z. Stejskal (State Inst. for Control of Drugs, Prague). *Českosl. Farm.*, 1957, **6** (3), 150–151.—The antihistamines of the *p*-chlorobenzhydrylpiperazine series (including buclizine, hydroxyzine and meclozine) were analysed by a chromatographic ion-exchange method. After extraction from pharmaceuticals with hot ethanol, the liberated base was titrated potentiometrically with 0·1 *N* HCl (antimony and calomel electrodes were used). The mean error was 3·5%. J. Volke

1684. **Gravimetric and polarographic determination of derivatives of xanthen-9-carboxylic acid (Banthine and Pro-Banthine).** J. Kráčmar and Z. Stejskal (State Inst. for Control of Drugs, Prague). *Československ. Farm.*, 1957, **6** (3), 139–141.—Methanthelinium bromide (Banthine bromide) and propantheline bromide (Pro-Banthine bromide) are pptd. by tungstosilicic acid (**I**) in acid soln. yielding a crystalline product of the composition $SiO_2.12WO_3.4B$, where B is the base. The polarographic titration is carried out at $-0{\cdot}65$ V in $\simeq 0{\cdot}5\ M$ HCl with $0{\cdot}01\ M$ **I**. The average error was $\pm 1\%$ for the drug and $\pm 2\%$ for the tablets (polarography), and $\pm 2\%$ for the drug and $\pm 3{\cdot}5\%$ for the tablets (gravimetry). J. VOLKE

1685. **Structural analysis of clinical dextrans by periodate oxidation and isotope dilution techniques.** J. D. Moyer and H. S. Isbell (Nat. Bureau of Standards, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1862–1865.—The method, for the determination of the 1:3′- and 1:4′-linkages of dextran, is based on the quant. determination of the fragments formed by periodate oxidation of the polysaccharide and hydrolysis of the resulting polymeric dialdehyde. D-Glucose and D-erythrose derived from anhydroglucose with 1:3′- and 1:4′-linkages, respectively, were determined by reaction with ^{14}C-labelled NaCN. The resulting nitriles were converted without isolation into radioactive aldonic acids, from which suitable salts were prepared and separated by co-crystallisation with non-radioactive carriers. The results were calculated by the usual procedure. The method was applied to five samples of clinical dextran and the results are listed. G. P. COOK

1686. **Volumetric method for the determination of bismuth in various compounds and pharmaceutical preparations. II.** M. B. Shchigol' and N. B. Burchinskaya. *Aptechnoe Delo*, 1957, **6** (2), 36–38.—The alkalimetric method for the determination of Bi (*Anal. Abstr.*, 1957, **4**, 1511) is applied to bismuth subnitrate and to various pharmaceutical preparations containing this compound. E. HAYES

1687. **Improved technique for toxicological extractions.** P. L. Kirk and C. L. Brown (School of Criminology, Univ. of Calif., Berkeley, U.S.A.). *Mikrochim. Acta*, 1957, (5), 714–719 (in English).—Two designs of liquid - liquid double extractor have been used for the removal of small amounts of acidic or basic poisons from biological materials. Preliminary removal of protein, fat, etc., is not needed. In the more satisfactory design, two concentric compartments contain aq. phases in contact with a continuous immiscible solvent with $d > 1$. For the extraction of alkaloids, the aq. phase in the outer compartment is the sample at pH 9, that in the inner compartment is dil. HCl or H_2SO_4, and the lower layer is $CHCl_3$ or CCl_4. After magnetic stirring for 3 or 4 hr., the alkaloid is isolated in a high state of purity from the inner compartment; good recoveries (85%) are obtained by renewing the acid phase after extraction for 2 or 3 hr. Extraction of a barbiturate gave a pure product in low yield (25%). A similar apparatus for use with organic solvents with $d < 1$ is less satisfactory. A. R. ROGERS

See also Abstracts—**1514**, Determination of iodide and iodine in solution. **1553**, Detection of acid hydrazides and barbiturates. **1560**, Determination of methyl and ethyl salicylates. **1587**, Determination of acetylsalicylic acid, phenacetin and caffeine in mixtures. **1598**, Determination of isoniazid in biological fluids. **1599**, Determination of chlorpromazine in blood and urine. **1612**, Fluorescence of ethylenediamine derivatives of adrenaline and noradrenaline.

Food

1688. **Rapid method of sugar analysis suitable for control processes.** C. C. Strachan and J. A. Kitson. *Canad. Food Ind.*, 1956, **27** (11), 22–23.—The method is suitable for materials having a high sugar content and gives results approximating to those from the official A.O.A.C. method. A representative sample is ground and 50 g is extracted with 465 ml of water in a 1-litre-capacity Waring Blendor for 5 min. at 20°. After being filtered rapidly with fluted Whatman No. 4 paper, the soln. is cooled to 20°. To determine invert sugar, 20 ml of filtrate is made up to 500 ml with water at 20° and then titrated against 10 ml of mixed Fehling's soln. according to the Lane and Eynon volumetric method. To determine total sugar as invert, 20 ml of the filtrate is pipetted into a flask containing 5 g of citric acid crystals and 80 ml of water. The soln. is gently boiled for 10 min. and cooled, it is then exactly neutralised with a 10% NaOH soln., with phenolphthalein as indicator. After cooling to 20° this neutral soln. is made up to 200 ml with water at 20° and titrated against 10 ml of the Fehling's soln. The sucrose content is estimated as the difference between total sugar and invert sugar content. The percentage of sugar is calculated by dividing 25 × a factor [the factor varies with the titre and is found from a table —"A.O.A.C. Methods of Analysis," 1955, p. 906] by the titre. If the sugar content is 13 to 42%, the 20-ml aliquot is diluted to 250 ml and the factor is then multiplied by 12·5. Tables of results are given showing the percentage of sugar estimated by this method and by the official method.

SUGAR IND. ABSTR.

1689. **Marc contents of sugar beet.** K. Vukov. *Növénytermelés*, 1956, **5**, 355–366.—A quick method for determining marc in beet comprises the taking of a sample of pulp from the beet by means of a segment rasp, drying the pulp and determining the total dry-solids content, making an aq. extract of the same pulp, and determining the soluble dry-solids content by refractometer. The difference between the two solids contents represents the insoluble dry solids, *i.e.*, the marc. The error of the method is $\pm 0{\cdot}39\%$, due largely to the deficiency of the refractometric measurement. A high correlation (0·79) was found between the hydrated marc volume (t ml/100 g) and the dry marc content (m g/100 g), the regression equation being $t = 2m - 3{\cdot}5$. There was only a slight correlation (0·41) between the resistance to cutting of the beet and the marc content, but the correlation of cutting resistance with crude fibre is 0·52. The workability of the beet is thus affected more by the composition of the marc than by the amount of marc.

SUGAR IND. ABSTR.

1690. **The quantitative determination of amino acids in foodstuffs. Potato albumin.** A. Kastell (Landwirtschaftlich-chem. Bundesversuchsanstalt, Wien). *Mikrochim. Acta*, 1957, (5), 689–698 (in German).—A paper-chromatographic method described for the quant. determination of the amino

acids in foodstuff hydrolysates. The solvents and buffers used by McFarren (*cf. Brit. Abstr. C*, 1951, 217) are employed in uni-dimensional development, and colorimetry is performed on the products of reaction with $Cu(NO_3)_2$ or ninhydrin. The accuracy is estimated to be $\simeq \pm 5\%$. A. R. ROGERS

1691. **Analysis of tissue constituents. Commercial ox-muscle extract.** T. Wood and A. E. Bender (Res. Dept., Bovril Ltd., 148–166 Old St., London, England). *Biochem. J.*, 1957, **67** (3), 366–373.—A scheme is suggested for the separation, identification, and determination of the compounds in muscle extracts, and it is applied to commercial meat extract. Proteins are separated by dialysis and inorganic salts by electrodialysis. The substances present in the extract are identified by paper chromatography and a range of colour reagents. R_F values of compounds likely to be present in such extracts are given; they include those for a wide range of peptides, organic acids, organic phosphates, guanidines, quaternary ammonium compounds, purines, pyrimidines and inorganic ions. A new process is described for the identification of organic acids. It depends on a preliminary separation by electrodialysis followed by two-dimensional paper chromatography. When identified, the various constituents are determined by standard methods or by direct estimation of the colour intensity on their chromatograms. The wt. of colouring matter is determined by deposition on ion-exchange membranes after electrodialysis. J. N. ASHLEY

1692. **Quantitative ionophoretic determination of whey proteins in skim milk.** A. Leviton (Eastern Utilization Res. Branch, Agric. Res. Service, U.S. Dep. Agric., Wash., D.C.). *J. Agric. Food Chem.*, 1957, **5** (7), 532–538.—Modifications in technique in applying paper-electrophoretic methods to the quant. study of whey proteins are discussed. In studies with α-lactalbumin and β-lactoglobulin, singly and in mixtures, recovery was quant. if a soln. of β-lactoglobulin was applied to the paper strip and submitted to electrophoresis before the application and electrophoresis of the sample. Details are given. When applied to whey, this pre-coating technique gave quant. values for the chief protein components. Streaking of the sample 1 in. from the apex towards the cathode resulted in better resolution of β-lactoglobulin in whey and skim milk. S.C.I. ABSTR.

1693. **Organic acids in raw and heated skim milk.** C. V. Morr, W. J. Harper and I. A. Gould (Ohio Agric. Exp. Sta., Wooster, U.S.A.). *J. Dairy Sci.*, 1957, **40** (8), 964–972.—Chromatography is on two-section columns of silicic acid treated with 0·5 *N* H_2SO_4. Development with butanol - chloroform mixtures gives acidity peaks in the eluate for butyric, propionic, acetic, pyruvic, formic, lactic and four unidentified acids. The variations in total and individual acids are discussed in relation to different samples, the amount of heating and browning produced during heating (100°, for $\gtrless 8$ hr.). W. H. C. SHAW

1694. **Determination of copper in industrial curd [for cheese].** V. Malý (Průmysl Škola Mlékárenská, Kroměříž, Czechoslovakia). *Průmysl Potravin*, 1957, **8** (4), 201–202.—The reduced form of phenolphthalein is oxidised in the presence of Cu^{2+}, and the resulting colour is measured. *Procedure*—To the sample (10 g) add H_2O_2 (3%) (0·5 ml) and 3 ml of phenolphthalein soln. (7 g of phenolphthalein dissolved in 150 ml of H_2O and 30 g of KOH, to which is added 10 g of zinc dust and, with warming, 20 g of $SnCl_2$). The resulting colour is compared with standards. As little as 0·00005% of Cu can be detected. J. ZÝKA

1695. **Determination of oxidation changes of fats by a colorimetric method using 2-thiobarbituric acid.** B. Sedláček and R. Rybín (Inst. Hyg., Prague, Czechoslovakia). *Průmysl Potravin*, 1957, **8** (1), 44–45.—A mixture of the sample (5 g), H_2O (100 ml) and 3 *N* HCl (5 ml) is distilled, and to a 20-ml aliquot of the distillate are added thiobarbituric acid soln. (1%) (1 ml) and H_3PO_4 (85%) (1 ml). The product is heated on a water bath for 35 min., and the resulting pinkish-red colour is measured at 530 mμ. J. ZÝKA

1696. **Fluoriphotometric analysis of fats.** F. Provvedi (Lab. Chim. Prov. di Bergamo, Italy). *Olii Min.*, 1957, **34** (8), 373.—Decolorised soln. of the fats (25%) in benzene are examined in a fluoriphotometer and the fluorescence intensities are expressed as a percentage of that given by a standard quinine sulphate soln. Butter gives a low intensity (14 to 19%) and may readily be differentiated from margarine (25 to 53%). Indications of adulteration may also be obtained. Cacao butter (9%) may be distinguished from coconut butter (20%) and hydrogenated arachis oil (33·5%). L. A. O'NEILL

1697. **A note on the separation of saturated fatty acids by circular paper chromatography.** T. Barnabas and J. Barnabas (Dept. of Chem., Ahmednagar Coll., India). *Naturwissenschaften*, 1957, **44** (9), 281–282.—The circular paper discs are impregnated with liquid paraffin in benzene (3:20) by a chromatographic run with this mixture in the apparatus for 4 hr. On removal from the apparatus the paper is dried and ready for use. For developing, either glacial acetic acid alone or with an admixture of 5 or 10% of benzene is used. The developing solvent is saturated with liquid paraffin in a separating funnel and the lower phase is used for development. The locating reagent is 2% aq. Pb acetate and a satd. soln. of aq. H_2S. E. KAWERAU

1698. **Quantitative mass-spectrometric analysis of mixtures of unsaturated and saturated fatty acids.** B. Hallgren, E. Stenhagen and R. Ryhage (Inst. of Med. Chem., Univ. of Uppsala, Sweden). *Acta Chem. Scand.*, 1957, **11** (6), 1064–1065.—High-resolution mass spectrometry of methyl esters of long-chain saturated and unsaturated carboxylic acids shows a large parent peak due to unfragmented ionised parent molecules, and these peaks may be used to analyse mixtures of esters. The sensitivity coefficients of the parent peaks differ somewhat, so that empirical corrections must be applied. The parent peaks for methyl linoleate at $m/e = 294$ and for methyl linolenate ($m/e = 292$) are accompanied by a peak at $m/e = 290$, which is thought to be the parent peak of a C_{18}-acid methyl ester with four double bonds, apparently present as an impurity in the original sample. E. J. H. BIRCH

1699. **The determination of vitamin A in cod-liver oil.** S. Erbe (Pharmac. Inst., Univ. Halle, Germany). *Pharm. Zentralh.*, 1957, **96** (12), 611–620.—A review is presented of methods of determination of vitamin A, including the growth test, the xerophthalmia test and the kolpokeratosis test (Moll *et al.*, *Arch. exp. Path. Pharmak.*, 1933, **170**, 76) which is preferred and described in detail.

The colour tests with $SbCl_3$ and with 3:3-dichloropropane-1:2-diol and the spectrophotometric method are also considered. S. BAAR

1700. **Studies on vitamin B_1 and related compounds. LXXX. Heat decomposition of disulphide-type thiamine derivatives. (1). Separate determination of thiochrome, thiamine thiazolone and thiothiamine.** Shojiro Yurugi (Res. Lab., Takeda Pharm. Ind., Juso-nishinocho, Higashiyodogawa-ku, Osaka). *J. Pharm. Soc. Japan*, 1957, **77** (1), 19–22.—Thiochrome (**I**), thiamine thiazolone (**II**), and thiothiamine (**III**), decomposition products of disulphide-type thiamine, are separated by paper chromatography (*Ibid.*, 1954, **74**, 502). The spots corresponding to R_F 0·4 to 0·45 (**I**), 0·6 and 0·7 (for **II** and **III**) are cut out and each is extracted with 0·02 *M* acetate buffer (pH 3·7) (5 ml). After over-night refrigeration the soln. is filtered and submitted to photometry at 394 mμ (**I**), 245 mμ (**II**) and 323 mμ (**III**). The spot at R_F 0·6 contains some **III** and the amount is added to that obtained at R_F 0·7. For amounts of **II** and **III** >30 μg the extraction is incomplete and the sample soln. should be diluted. K. SAITO

1701. **A further study of the determination of thiamine with 6-aminothymol.** K. J. Hayden and R. H. Elkington (Novadel Ltd., St. Ann's Crescent, Wandsworth, London). *Analyst*, 1957, **82**, 650–652.—An investigation of the method for determining thiamine by measurement of the colour produced with diazotised 6-aminothymol (Hayden, *Analyst*, 1957, **82**, 61) has shown that diazotisation is not necessarily involved in the reaction, and that a similar colour is produced by direct treatment of the vitamin with an alkaline soln. of 6-aminothymol. The vitamin is extracted with saturated KCl soln., but if $CaCO_3$ is present in the form of precipitated chalk the extraction can be made satisfactorily with water. Samples containing metals, metal salts or other substances reacting with thiamine in acid soln. are treated with enough precipitated chalk to neutralise subsequently any free acid, and are then extracted with water, and the filtered extract is adjusted to contain 20 to 40 μg of thiamine per ml. Samples containing creta praeparata are extracted with saturated KCl soln. and the filtered extract is adjusted as before. Preparation of the alkaline 6-aminothymol is described, and the general method of determination is as previously described (*loc. cit.*). Agreement between results with the original and modified methods is good. A. O. JONES

1702. **Ultra-violet absorption spectra of L-ascorbic acid in aqueous solutions.** J. S. Lawendel (Res. Centre, Italfarmaco S.p.A., Piazza Coriolano 5, Milan). *Nature*, 1957, **180**, 434–435.—New values are given for the extinction coeff., E (1%, 1 cm), of non-dissociated and mono-dissociated L-ascorbic acid, namely 695 at 245 mμ (pH 2 with HCl soln.) and 940 at 265 mμ (pH 6·4 with phosphate buffer). The results were obtained with final concn. ranging from 0·5 to 1 mole × 10^{-4}. Strict anaerobic conditions were used without chemical inhibitors. G. S. ROBERTS

1703. **Determination of dehydroascorbic acid by paper chromatography.** J. Davídek and V. Šanda (Res. Inst. Food Technol., Prague). *Českosl. Farm.*, 1957, **6** (3), 151–153.—Dehydroascorbic acid is determined by the difference between the analyses of a non-reduced sample and a sample reduced with H_2S. Quant. paper chromatography is carried out with the system butanol - acetic acid - water (4:1:5), and the spots of ascorbic acid are detected with 2:6-dichlorophenolindophenol. The spot areas of analysed samples are compared with those of control spots (with added ascorbic acid). J. VOLKE

See also Abstracts—**1440**, Stability of vitamin B_{12} in dry cereals. **1547**, Ultra-micro determination of fructose. **1548**, Micro-determination of fructose. **1596**, Determination of chloride in foods. **1661**, Determination of riboflavine.

Sanitation

1704. **Determination of radon and thoron in air.** G. B. Courtier (Public Health Dept., County Hall, London). *Nature*, 1957, **180**, 382–383.—Expressions are given that relate the concn. of radon in the atmosphere with the observed activity from mixed decay products in the filtration type of determination. Similar expressions could be formulated for thoron; owing to the long life of Th-B, thoron can be estimated in the presence of excess of radon. J. H. WATON

1705. **Determination of aromatic hydrocarbons in polluted air.** J. F. Thomas, B. D. Tebbens, M. Mukai and E. N. Sanborn (Univ. Calif., Berkeley, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1835–1840.—The particulate organic component was separated into three broad classes by conventional techniques involving solubility characteristics. Each group was further separated by paper-strip chromatography or electrophoresis into pure components which were identified by measuring u.v. absorption and fluorescent spectra directly from the paper. G. P. COOK

1706. **Measurement of concentrations of gaseous halide tracers in air by positive-ion-emission techniques.** H. A. Schultz (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1840–1842.—The odourless, colourless, non-corrosive and non-toxic gas, dichlorodifluoromethane (Freon 12), can be used as a tracer for studying stack effluents in the atmosphere. The gas can be measured in air at a concn. as low as 0·1 p.p.m. by special techniques involving the use of a modified halogen leak detector. K. A. PROCTOR

1707. **Determination of aerosol size distributions by jet impactor - light scattering technique.** J. K. Thompson (Naval Res. Lab., Washington 25, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1847–1850.—The aerosol is drawn from a sampling probe through a series of jet impactors with a range of characteristic diameters. The fraction of aerosol particles remaining airborne after passing through any particular impactor is then measured by a light-scattering meter. In this way, size-distribution data in terms of scattered-light intensity are obtained. K. A. PROCTOR

1708. **Improvements relating to the determination of small amounts of silver in water.** Equipro Corporation [Inventor: W. E. Chesney]. British Patent 788,211; date appl. 24.2.56.—Small amounts of silver, such as are used in water purification, are determined by filtering the sample (500 ml) through a membrane filter, to remove colloidal and extraneous matter, acidifying with dil. HCl, adding KI, and collecting the AgI on a restricted area (1 sq. cm) of a membrane filter, *e.g.*, a Lovell Millipore filter (type HA-plain). The AgI is converted into Ag_2S by the addition of Na_2S, and the

coloured disc is compared with standards prepared in a similar manner. J. M. JACOBS

1709. **Principles and problems in development of a dissolved oxygen analyser.** H. S. Levine and R. S. Kleinschmidt (Robert A. Taft Sanitary Engng Center, USPHS, Cincinnati, Ohio, U.S.A.). *Sewage Ind. Wastes*, 1957, **29**, 856–863.—A gasometric method for obtaining a continuous record of the dissolved oxygen content of water is described (*cf.* Levine *et al.*, *Anal. Abstr.*, 1956, **3**, 2569). S.C.I. ABSTR.

1710. **Determination of the chemical oxygen demand of citrus waste waters.** R. R. McNary, M. H. Dougherty and R. W. Wolford (Florida Citrus Exp. Sta., Lake Alfred, U.S.A.). *Sewage Ind. Wastes*, 1957, **29**, 894–900.—The procedure of Moore *et al.* (*Anal. Chem.*, 1949, **21**, 953; 1951, **23**, 1297) was studied with citrus waste water and it was shown that the min. reflux time could be reduced to a standardised 10 min. S.C.I. ABSTR.

See also Abstract—**1499**, Determination of traces of selenium in air and water.

Agriculture and Plant Biochemistry

1711. **A rapid method for the determination of calcium and magnesium in plant material by titration with disodium ethylenediaminetetra-acetate.** V. P. Padhye (Gov. Chemist's Dept., Dar Es Salaam, Tanganyika). *Analyst*, 1957, **82**, 634–638.—The dried, ground plant material is ignited with the aid of ethanol, and the residue is heated with $NaNO_3$, at first gently and finally to whiteness at 550°. The ash is digested with hot 6 *N* HCl, the digest is evaporated to dryness and heated to 110° to dehydrate the silica. The residue is dissolved in dil. HNO_3, the soln. is filtered, the ppt. is washed with dil. HNO_3 and the filtrate is adjusted to 25 ml. An aliquot is treated in a centrifuge tube with 1 ml of a 2% $Zr(NO_3)_4$ soln. to remove PO_4^{3-}, warmed, and centrifuged, and the supernatant liquid (with one washing) is treated with 1 ml of 1% Na diethyldithiocarbamate soln. and extracted repeatedly with CCl_4 to remove Fe and Mn complexes. The filtered aq. layer is adjusted to 25 ml and Ca is determined in an aliquot, after addition of KOH soln. and murexide indicator, by titration with standard EDTA (disodium salt) soln. Another aliquot is mixed with 5 ml of a buffer soln. at pH 10 (aq. NH_3 - NH_4Cl) and titrated for Mg with Eriochrome black T as indicator, the titre equiv. to Ca being deducted. Recovery of added Ca and Mg was good. A. O. JONES

1712. **Quantitative determination of carbonyl compounds and secondary and tertiary alcohols in small samples of plant material.** P. A. Kolesnikov (A. N. Bakh Inst. of Biochem., Acad. Sci. USSR, Moscow). *Biokhimiya*, 1957, **22** (4), 622–625.—The methods described are suitable for use with plant materials containing oil of coriander or oil of peppermint. The determination of carbonyl compounds is based on the reaction with 2:4-dinitrophenylhydrazine followed by absorptiometry in a photo-electric instrument with a green filter. The determination of alcohols depends on their reaction with *p*-dimethylaminobenzaldehyde in H_2SO_4 - H_2O (1·6:1, v/v). For the removal of interfering substances, 0·5 g of plant material is ground with 2·5 ml of a 5% soln. of trichloroacetic acid, and 7·5 ml of a 1% soln. of Ba acetate in methanol is added to the homogeneous suspension. The mixture is again ground and the liquid is filtered through paper. The excess of Ba is removed by adding a drop of dil. H_2SO_4 and filtering or centrifuging. Because this procedure does not remove completely substances that interfere in the alcohol determination, a blank containing 1 ml of test soln. plus 5 ml of dil. H_2SO_4 is used in the photometric evaluation of the colour produced by *p*-dimethylaminobenzaldehyde. The methods can be applied to a single plant-leaf. E. HAYES

1713. **Radial paper chromatography. Application to analysis of mixtures of chlorophyll derivatives.** M. J. Hendrickson, R. R. Beruefly and A. R. McIntyre (Univ. Nebraska, Omaha, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1810–1815.—A simple chromatographic apparatus of the radial type is described for the precise identification and determination of organic pigments soluble in non-polar solvents. By the use of statistical methods, two constants were derived, which related the movement of chlorophyll derivatives to the movement of solvent; both constants were useful for the identification of these derivatives. Data for 18 are listed. G. P. COOK

1714. **Correction of interference effects in the photometric determination of sodium, potassium and calcium in soil.** F. Burriel-Martí, J. Ramírez-Muñoz and A. Benito-Potous (Inst. Edafologia, C.S. de I.C., Madrid). *An. Real Soc. Esp. Fis. Quim, B*, 1957, **53**, 521–530.—Interference effects of soln. containing two elements have been studied to find suitable corrections for experimental values for Na, K and Ca in ammonium acetate extracts of soil containing these elements. The soln. used contained *N* ammonium acetate as background. Bilogarithmic graphs have been plotted of the concn. of the element being determined as a function of the concn. of interfering elements; hence the variations of the interference ratios have been calculated. Correction methods for the analysis of soil extracts are given. C. A. FINCH

1715. **Volumetric determination of sulphates in soil and irrigation water.** A. G. Ashgar, M. A. Qayyum and G. M. Rana (Land Reclamation Directorate, Pakistan). *Soil Sci.*, 1957, **83**, 239–241.—The water or soil extract (10 ml) is titrated with 0·02 *N* EDTA (disodium salt), with Eriochrome black T F-241 indicator. To a second sample (10 ml) is added 5 ml of 0·025 *N* $BaCl_2$ and 20 drops of a buffer soln. (67·5 g of NH_4Cl and 570 ml of conc. aq. NH_3 per litre). Titration with EDTA is carried out as before except that a few drops of 0·01 *N* KCN are added with the indicator. The difference between the two titrations (excess of Ba) serves to calculate the sulphate pptd. A. G. POLLARD

1716. **Iodimetric determination of iron in soil by the Mohr - Treadwell method.** M. A. Vinnik and E. N. Konnova. *Vestn. Moskov. Univ.*, 1956, (1), 89–93; *Ref. Zhur., Khim.*, 1957, Abstr. No. 54,707.—The applicability of the simple and rapid Mohr - Treadwell method to the determination of Fe^{3+} in soils is indicated. To 25 or 50 ml of the sulphate filtrate after removal of SiO_2, add 0·25 to 25 milliequiv. of HCl in a 250-ml conical flask. Add 10 ml of a freshly prepared 10% KI soln. or 1 g of KI and titrate the liberated iodine with 0·1 *N* or 0·02 *N* $Na_2S_2O_3$ in the presence of starch as indicator.

The deviation of the results from those of the Hahn - Windisch method is, in most cases, $< 5\%$.
C. D. KOPKIN

1717. **Polarographic and photocolorimetric micro-determination of copper, lead, nickel, zinc, cobalt and manganese in soil.** A. Duca and D. Stănescu (Inst. Chim., Acad. R.P.R., Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1957, **8** (1–2), 75–83.—Elements that are present in the order of 0·001% are estimated according to the following scheme. Approximately 5 g of the soil sample is disintegrated with $HClO_4$ (70%) or aqua regia, and treated finally with $HClO_4$. An aliquot of the filtrate is used for the colorimetric determination of Mn as $HMnO_4$. To the remainder of the soln. is added conc. aq. NH_3, to a pH of 9, and the resulting soln. is extracted with a soln. of dithizone in $CHCl_3$ (0·03%). The extracted cations are freed from their dithizonates and excess of dithizone by the addition of $HClO_4$ (70%) and H_2O_2 (30%), and the mixed ppt. are re-dissolved in HCl, and an aliquot is taken for the colorimetric determination of Pb. The remainder of the HCl soln. is evaporated to dryness, neutralised with aq. NH_3 and used for the polarographic determination of Cu, Ni and (Co - Zn), after which the Co is determined colorimetrically. An error of $< 10\%$ is claimed.
H. SHER

1718. **Rapid method for the determination of total exchange alkaline-earth bases in carbonate-free black earth.** P. V. Madanov and L. M. Voikin. *Pochvovedenie*, 1956, (12), 80–82; *Ref. Zhur., Khim.*, 1957, Abstr. No. 44,893.—The method is based on the irreversible reaction between the alkaline-earth bases of the soil and K oxalate, with the formation of the insol. Ca and Mg oxalates. To 10 g of soil add 250 ml of 0·1 *N* K oxalate, shake on an electric shaker for 1 hr. and set aside for 24 hr., with occasional shaking. Filter and to 50 ml of the filtrate add 10% H_2SO_4 (20 ml) and activated carbon (0·3 g), heat to boiling-point, filter, wash the beaker out with hot 10% H_2SO_4 and wash the filter 2 or 3 times with the same soln. Heat the clear filtrate to boiling-point and titrate the excess of K oxalate with 0·1 *N* $KMnO_4$.
C. D. KOPKIN

1719. **Gravimetric method for the determination of phosphate ion [in fertilisers] as lead phosphate.** M. Vancea and M. Voluşniuc (Inst. Chim., Acad. R.P.R., Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1957, **8** (1–2), 93–96.—The method is based on the pptn. of PO_4^{3-} as $Pb_3(PO_4)_2$ from a boiling aq. soln., in the presence of acetic acid - Na acetate buffer (pH 2·3), by 0·05 *N* Pb acetate. The results do not differ significantly from those obtained by the molybdophosphate method.
H. SHER

1720. **Direct volumetric determination of phosphate [in fertilisers].** **I.** M. Vancea and M. Voluşniuc (Inst. Chim., Acad. R.P.R., Cluj, Romania). *Stud. Cercet. Chim., Cluj*, 1957, **8** (1–2), 85–88.—The PO_4^{3-}, in the presence of an acetic acid - Na acetate buffer (pH 2 to 3) and a chloroform soln. of dithizone, are pptd. as $Pb_3(PO_4)_2$ by titration with Pb acetate. Any excess of Pb^{2+} forms a coloured complex with the dithizone, which thus serves as an internal indicator. An accuracy equal to that of the magnesium Eriochrome black T method is claimed.

II. M. Vancea and M. Voluşniuc. *Ibid.*, 1957, **8** (1–2), 89–92.—The procedure followed is similar to that in Part I, but with the use of a soln. of $Bi(NO_3)_3$ in dil. HNO_3 as titrant.
H. SHER

1721. **Sampling crops for residue analysis.** L. Lykken, L. E. Mitchell and S. M. Woogerd (Shell Chem. Corp., New York, N.Y., U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (7), 501–505.—The fundamental requirements for establishing the validity of any pesticide-residue analysis are reviewed, the first step being to obtain a truly representative sample. This depends on the careful design of the experiment, *viz*, establishing plots of adequate size, providing enough replicates and taking care to ensure uniform distribution of the pesticides. Careful collection, preparation, and analysis of samples then yield the max. of reliable data.
S.C.I. ABSTR.

1722. **Determination of the gamma isomer of hexachlorocyclohexane by measurement of the initial crystallisation point.** E. Degeorges and P. Mugnier. *Chim. et Ind.*, 1957, **77** (4), 794–797.—The method utilises the variation of the b.p. of a pure liquid (toluene) with pressure. The toluene is contained in a thermostatic jacket surrounding a freezing tube, fitted with a condenser to which a mercury manometer is connected. The b.p. of the toluene is raised to 116° by raising the reservoir of the manometer, and the product to be tested (which has been maintained at 140° for 30 min. with stirring and then cooled to 120°) is poured into the freezing tube so that the bulb of the Beckmann thermometer is completely immersed. When the temp. of the molten product and the toluene in the jacket have reached equilibrium ($\simeq$ 5 min.) stirring is begun at the rate of $\leqslant$ 2 back-and-forth movements per sec. The jacket is restored to normal pressure and the temp. is read every 30 sec., the rate of cooling being 0·4° per min. At 0·1° below the crystallisation temp. the melt is seeded with a small amount of the product under test which has crystallised in its stable form on the walls of the beaker in which it was melted. Supercooling may amount to 0·4° and is followed by a rise in temp., the plateau being indicated by 4 or 5 readings at the same temp. A second determination is then made and should not differ by more than 0·01° from the first, otherwise a third determination must be made and only the concordant results retained. A lowering of the point of initial crystallisation by 0·01° corresponds to 0·018% of impurities. The method gives accurate results on products of 99·8% purity.
J. M. JACOBS

1723. **Colorimetric micro-determination of the acaricide 4:4′-dichloro-α-(trichloromethyl)benzhydrol (FW-293) [2:2:2-trichloro-1:1-di-(*p*-chlorophenyl)ethanol] [Kelthane].** I. Rosenthal, G. J. Frisone and F. A. Gunther (Rohm & Haas Co., Philadelphia, Pa.). *J. Agric. Food Chem.*, 1957, **5** (7), 514–517.—Under mildly alkaline conditions, FW-293 yields 4:4′-dichlorobenzophenone and chloroform (**I**). The presence of **I** was confirmed by gas chromatography and i.r. examination. A method for determining **I** based on the Fujiwara reaction was developed, in which **I** in a pyridine - water - NaOH mixture is heated to 100° to produce a red dye (absorption max. 530 and 364 mμ; mol. extinction coeff. at 530 mμ, 14,150, in aq. pyridine). The special apparatus required to sweep the liberated **I** from the reaction mixture is described and illustrated. Under optimum conditions, the method responds to 10 μg of FW-293 with 99% efficiency in the presence of various fruit and nut extractives.
S.C.I. ABSTR.

1724. **Ultra-violet spectrophotometric micro-determination of the acaricide 4:4′-dichloro-α-(trichloromethyl)benzhydrol (FW-293).** F. A. Gunther and R. C. Blinn (Univ. of California Citrus Expt. Sta., Riverside, U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (7), 517–519.—After alkaline hydrolysis of the acaricide FW-293, the *pp′*-dichlorobenzophenone moiety of the mol. may be determined quant. by its absorption at 264 mμ (mol. extinction coeff. value in 95% ethanol, 21,540 at 264 mμ), or by the absorption of the readily formed 2:4-dinitrophenylhydrazone in alcoholic alkali at 510 mμ. In application to residues on oily or waxy substrates, chromic anhydride oxidation of extraneous extracts is recommended. Under optimum conditions the method responds to 10 μg of FW-293 with 84% efficiency in the presence of citrus peel extractives. S.C.I. Abstr.

See also Abstracts—**1499**, Determination of traces of selenium in soil. **1569**, Analysis of toxic phosphorus compounds.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1725. **Proceedings of the National Conference on Instrumental Methods of Analysis. Chicago. June 13–15, 1957.** Publ. by Instrument Society of America (313 Sixth Avenue, Pittsburgh, Pa., U.S.A.). The following communications of analytical interest are included. **Use of analysis instrumentation in process control,** A. R. Aikman, 13 pp. **Neutron absorptiometry,** D. D. Deford and R. S. Braman, 9 pp. **Optical methods of process stream analysis,** L. G. Glasser, 10 pp. **A third of a century of X-ray analysis in industry,** G. L. Clark, 6 pp. **Applications of servo systems to the remotely operated chemical analysis of extremely radioactive solutions,** M. T. Kelley, D. J. Fisher, R. W. Stelzner, C. L. Burros, E. B. Wagner and H. L. Hemphill, 11 pp. **Surface analysis with a 2-million-volt Van de Graaff,** S. Rubin, T. O. Passell and L. E. Bailey, 11 pp. **The galvanic cell oxygen analyser,** W. J. Baker, J. F. Combs, T. L. Zinn, A. W. Wotring and R. F. Wall, 12 pp. **Nuclear magnetic resonance—a new tool for moisture analysis,** H. Rubin, 10 pp. **Characteristics of a polarographic mercury membrane electrode,** R. C. Bowers and A. Wilson, 3 pp. **Multi-component photo-electric analysis,** D. J. Troy, 7 pp. **Ultra-violet applications of Ulvir analyser,** M. H. Axler, 4 pp. **Hydrocarbon measurements in drilling muds,** F. B. Gray, jun., 8 pp.

1726. **Combustion micro-analysis of volatile liquids.** J. M. Corliss (Chem. Warfare Lab., Army Chemical Center, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1902.—An apparatus for micro-Dumas nitrogen determinations is described. E. G. Cummins

1727. **Automatic stopcock twister.** H. V. Malmstadt (Dept. of Chem. and Chem. Engng, Univ. of Illinois, Urbana, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1901.—The device described can be used not only on stopcock burettes that are used with automatic titrimeters, but also on stopcocks on any apparatus, especially where remote control of a series of stopcocks is needed. It can be used with lubricated glass or non-lubricated Teflon stopcocks and is convenient for manual titrations. K. A. Proctor

1728. **Ultra-micro chelometric titrations with potentiometric end-point detection.** F. S. Sadek and C. N. Reilley (Univ. of N. Carolina, Chapel Hill, N.C., U.S.A.). *Microchem. J.*, 1957, **1** (2), 183–201.—A mercury electrode in contact with a soln. containing metal ions and added Hg^{II} - EDTA acts as a half-cell *vs.* the S.C.E. Its use is described for the potentiometric titration of several metal ions, in concn. as low as 10^{-5} *M* and amounts of <22 μg, with 0·01 *M* EDTA (disodium salt) as titrant. A suitable cell is illustrated and the procedure is described, together with the precautions to be taken. Halides interfere, and the calomel electrode must be made up with KNO_3 in place of the more usual KCl. Interference by O in alkaline media is removed by bubbling N after each addition of titrant. End-points are obtained from a plot of vol. of titrant added *vs.* mV, in the usual way. Results are given for Cu, Pb, Ca, Ba, Ni, Co, La, Bi, Hg and Al. Coefficients of variation are $\simeq \pm 1\%$ or less in the 22-μg region, rising to $\pm 2\%$ or more in the 2-μg region. The possibility of the selective titration of one metal in presence of others, by the use of other chelating agents, is discussed. R. E. Essery

1729. **Laboratory apparatus for the recovery of small amounts of solute from dilute aqueous solutions.** M. T. Bush (Vanderbilt Univ. School of Medicine, Nashville, Tenn., U.S.A.). *Microchem. J.*, 1957, **1** (2), 269–274.—All-glass apparatus, fitted with standard taper joints, is illustrated, in which the solute is continuously extracted by distillation of solvent, and accumulates in the boiling flask. The apparatus can be used for batches of soln. from 50 ml to 5 litres, and can be adapted for solvents lighter or heavier than water. Performance was not specially studied, but 129 mg of barbitone was recovered out of 223 mg from 5 litres of soln. by continuous extraction with 250 ml of 1:2-dichloroethane for 24 hr. This recovery would be attained by 22 batchwise extractions with 250-ml portions of solvent, or 7 batchwise extractions with portions of 1 litre. Suggestions are made for improvement in design and for dealing with emulsification. R. E. Essery

1730. **Simple apparatus for countercurrent distribution.** O. Wichterle and O. Mikeš (Lab. Macromol. Chem., Chem. Inst. Acad. Sci., Prague). *Chem. Listy*, 1957, **51** (8), 1569–1574.—A detailed description is given of a new simple apparatus, the efficiency of which was determined on a mixture of three dyes with different distribution coefficients. The degree of the distribution was assessed colorimetrically, with the use of a single wavelength. J. Zýka

1731. **Automatic apparatus for the quantitative micro-determination of nitrogen.** F. E. Charlton (Min. of Supply, Chem. Defence Expt. Estab., Porton, Wilts., England). *Analyst*, 1957, **82**, 643–648.—The apparatus is described in detail. The method is similar to the micro-Dumas method and gives equally good results. It has the advantage that time is saved and that it does not require the attention of the operator during the whole period of the determination. Results with micro-analytical standards and with several organic compounds are quoted and indicate satisfactory accuracy. A. O. Jones

1732. **Arrangement for the application of samples for paper chromatography.** B. Meloun and O. Mikeš (Biochem. Dept., Chem. Inst. Acad. Sci., Prague). *Chem. Listy*, 1957, **51** (8), 1574–1575.—A simple arrangement is described for the acceleration of the application of samples for paper chromatography. J. ZÝKA

1733. **A simple aid for obtaining equilibration of the paper with the vapour phase of the solvent in chromatographic technique.** A. Ensgraber (Marburg, Hyg. Inst. der Univ., Aussenstelle Fulda, Germany). *Naturwissenschaften*, 1957, **44** (9), 281.—This method is particularly suitable for paper cylinders intended for ascending development. The paper cylinder is held in suspension above the solvent in the closed vessel for any desired length of time by the following arrangement. A cross-bar of thin metal (iron) sheeting is fixed to the top of the paper cylinder and a magnet to the outside lid of the glass jar. The magnet holds the paper above the solvent and, when it is desired to start the run, the magnet is withdrawn and the paper cylinder drops down into the solvent. E. KAWERAU

1734. **Compact apparatus for ascending chromatography.** W. R. Eberlein (Children's Hosp. of Philadelphia, Univ. of Pennsylvania, U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1957, **17** (12), 1476–1478.—The apparatus comprises a stainless-steel paper holder for use with a Pyrex-glass hydrometer jar, and is designed to accommodate a $9\frac{1}{4} \times 22\frac{1}{2}$ in. chromatogram. The method of use is described. H. F. W. KIRKPATRICK

1735. **New capillary viscometer.** N. N. Greenwood and K. Wade (Dept. Chem., Univ. of Nottingham, England). *J. Sci. Instrum.*, 1957, **34** (7), 288–289.—The instrument is suitable for measurement of viscosities of liquids that are unstable in air. It comprises a capillary tube sealed to a reservoir bulb into which the liquid is distilled. Inversion of the apparatus causes the liquid to flow from the reservoir bulb through a calibrated chamber (as in the Ostwald viscometer) and through the capillary. The lower tip of the capillary discharges into a weir which ensures that the level to which the liquid flows is constant. G. SKIRROW

1736. **Improved rotation viscometer for the study of low-viscosity liquids at low and intermediate rates of shear.** E. H. Frei, D. Treves and H. Eisenberg (Weizmann Inst. of Science, Rehovot, Israel). *J. Polym. Sci.*, 1957, **25** (110), 273–278.—An improved model of a precision rotation viscometer previously described (*J. Polym. Sci.*, 1954, **14**, 417) makes use of electrostatic restoring torque for the centipoise range. Torques from 0·1 to 500 dyne-cm are measured with an accuracy of 0·2%. Rates of shear range from 0·1 to 200 $sec.^{-1}$ B. J. WALBY

Optical

1737. **Device for the safe interchange of the lens and prism mounts of a Bausch and Lomb Littrow spectrograph.** W. H. Abbitt (Washington State College, Pullman, U.S.A.). *J. Opt. Soc. Amer.*, 1957, **47** (9), 862.—Accessories are described that enable the glass and quartz optical systems of a Littrow-type large spectrograph to be interchanged without appreciable risk of damage to the optical components. A special design of cradle is provided for each mount, which includes two handles for lifting but does not interfere with the proper seating of the mount in correct adjustment in the spectrograph. The other accessory is a box, attached to the side of the spectrograph, for housing the mount not in current use. B. S. COOPER

1738. **Diffusion cell scanning attachment for Beckman Model DU spectrophotometer.** E. Back, V. F. Felicetta and J. L. McCarthy (Univ. of Washington, Seattle, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1903–1904.—The attachment consists of a cell-holder, directly connected to a finely threaded, horizontal carrier screw. The latter is mounted in a screw housing, which also serves as a cover for the light-proof cell compartment of the spectrophotometer fitted with a photomultiplier tube attachment. The equipment can be used to measure the absorbance of materials that have diffused into a gel. K. A. PROCTOR

1739. **Artefact in spectrophotometry caused by fluorescence.** A. H. Mehler, B. Bloom, M. E. Ahrendt and D.-W. Stetton, jun. (Nat. Inst. of Arthritis and Metabolic Dis., Bethesda, Md., U.S.A.). *Science*, 1957, **126**, 1285–1286.—A cause of distorted spectra in the low u.v. region, occurring even when a double monochromator spectrophotometer is used, is illustrated and discussed. H. F. W. KIRKPATRICK

1740. **Some experiments with spectrofluorimeters and filter fluorimeters.** C. A. Parker and W. J. Barnes (Admiralty Materials Lab., Poole, Dorset, England). *Analyst*, 1957, **82**, 606–618.—Fluorescence emission spectra of liquids have been measured with laboratory-made accessories that could be combined in a versatile manner with a small monochromator, photomultiplier tube and a.c. amplifier. The apparatus can also be used for the measurement of fluorescence "excitation" spectra or as a simple filter fluorimeter for high sensitivity. The experiments described were designed to investigate the analytical possibilities of fluorescence spectrometry and to examine its limiting factors. Examples of the application of fluorescence emission spectra are given, and some general considerations of importance in quant. work are discussed in relation to the determination of sub-microgram amounts of boron by reaction with benzoin to form a fluorescent compound. A Dewar vessel for the measurement of low-temp. luminescence or absorption is described. A. O. JONES

See also Abstract—**1435**. Production of spark lines in a hollow cathode tube with pulse emission. A triggered a.c. spark for metal analysis. Design and performance of a fore-prism-grating i.r. spectrometer.

Thermal

1741. **Thermostat using a resistance thermometer and a galvanometer - photocell amplifier.** P. M. Hu and R. W. Parsons (Physics Dept., Univ. Hong Kong). *J. Sci. Instrum.*, 1957, **34** (7), 283–285.—A copper-wire resistance thermometer forms one arm of a Wheatstone network. Light reflected from the galvanometer mirror is deflected by a prism on to each of two photocells, which are connected in adjacent arms of a second Wheatstone bridge. The output from this bridge is fed into an amplifier (gain 5×10^5). The apparatus is capable of keeping the temp. constant to within $\pm 0{\cdot}005°$ at temp. up to 65°. G. SKIRROW

1742. **Description of a new type of Parr bomb.** M. J. Maurice and J. W. Y. Heynis (N. V. Onderzoekingsinst. "Research," AKU, Arnhem, Netherlands). *Mikrochim. Acta*, 1957, (5), 658–660 (in English).—A Parr bomb has been made from nickel-plated stainless steel, large enough for the destruction of 1-g samples of rayon yarn or rubber with Na_2O_2 (10 g). An aluminium gasket separates the bomb from the lid, which is tightened with the aid of a bayonet catch and a pressure screw. There is no leakage, provided that the melt does not touch the gasket or lid. A. R. ROGERS

1743. **Chromatothermographic universal apparatus for the analysis of complex gaseous mixtures.** N. M. Turkel' taub and A. A. Zhukhovitskiĭ (All-Union Sci. Res. Geological Prospecting Petroleum Inst.). *Zavod. Lab.*, 1957, **23** (9), 1120–1124. G. S. SMITH

1744. **Derivative thermometric titrations.** S. T. Zenchelsky and P. R. Segatto (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1856–1858.—The selection of the end-point in a thermometric titration is aided by an apparatus for obtaining first and second derivative curves. Mechanical amplification is used, with a filter to increase signal to noise ratio. K. A. PROCTOR

Electrical

1745. **Anodic-stripping voltammetry using the hanging-mercury-drop electrode.** R. D. DeMars and I. Shain (Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1825–1827.—In this rapid and convenient method of analysing dil. soln. of metals that form amalgams, the techniques of voltammetry are used with a continuously varying potential. The anodic peak current is a function of concn. of the ion in soln. and the cathodic plating time. The method has been applied to Cd and Th[1] in the 10^{-6} to 10^{-9} *M* range and to mixtures of Cd and Zn in the 10^{-4} to 10^{-6} *M* range. K. A. PROCTOR

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ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	milli-equivalent	milli-equiv.
ampere	amp.	milligram	mg
Ångström unit	Å	millilitre	ml
anhydrous	anhyd.	millimetre	mm
approximate, -ly	approx.	millimicrogram	mμg
aqueous	aq.	millimolar	m*M*
atmospher-e, -ic	atm.	millivolt	mV
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	*M*
calorie (large)	kg-cal.	molecul-e, -ar	mol.
calorie (small)	g-cal.	normal (concentration)	*N*
centimetre	cm	optical rotation	α_λ^t
coefficient	coeff.	ounce	oz
concentrated	conc.	parts per million	p.p.m.
concentration	concn.	per cent.	%
constant	const.	per cent. (vol. in vol.)	%(v/v)
corrected	(corr.)	per cent. (wt. in vol.)	%(w/v)
crystalline, crystallised	cryst.	per cent. (wt. in wt.)	%(w/w)
cubic	cu.	potential difference	p.d.
current density	c.d.	precipitate (as a noun)	ppt.
cycles per second	c/s	precipitated	pptd.
density	ρ	precipitating	pptg.
density, relative	*d* or wt. per ml	precipitation	pptn.
dilute	dil.	preparation	prep.
direct current	d.c.	qualitative, -ly	qual.
distilled	dist.	quantitative, -ly	quant.
ethylenediaminetetra-acetic acid	EDTA	recrystallised	recryst.
		refractive index	n_λ^t
electromotive force	e.m.f.	relative band speed	R_F
equivalent	equiv.	relative humidity	r.h.
gram	g	revolutions per minute	r.p.m.
gram-molecule	mole	saponification value	sap. val.
half-wave potential	$E_{\frac{1}{2}}$	saturated calomel electrode	S.C.E.
hour	hr.	second (time)	sec.
hydrogen ion exponent	pH	soluble	sol.
infra-red	i.r.	solution	soln.
insoluble	insol.	specific gravity	sp. gr.
international unit	i.u.	specific rotation	$[\alpha]_\lambda^t$
kilogram	kg	square centimetre	sq. cm
kilovolt	kV	standard temp. and pressure	s.t.p.
kilowatt	kW	temperature	temp.
liquid	liq.	ultra-violet	u.v.
maxim-um, -a	max.	vapour density	v.d.
melting-point	m.p.	vapour pressure	v.p.
microgram	μg (not γ)	volt	V
microlitre	μl	volume	vol.
micromole	μmole	watt	W
micron	μ	wavelength	λ
milliampere	mA	weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	>	less than	<
not greater than	≯	not less than	≮
is proportional to	∝	of the order of, approximately	≃

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{2+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

1742. **Description of a new type of Parr bomb.** M. J. Maurice and J. W. Y. Heyns (N. V. Onderzoekingsinst. "Research," AKU, Arnhem, Netherlands). *Mikrochim. Acta*, 1957, (5), 658–660 (in English).—A Parr bomb has been made from nickel-plated stainless steel, large enough for the destruction of 1 g samples of rayon yarn or rubber with Na_2O_2 (10 g). An aluminium gasket separates the bomb from the lid, which is tightened with the aid of a bayonet catch and a pressure screw. There is no leakage, provided that the melt does not touch the gasket or lid. A. R. ROGERS

1743. **Chromatothermographic universal apparatus for the analysis of complex gaseous mixtures.** N. M. Turkel'taub and A. A. Zhukhovitskiĭ (All-Union Sci. Res. Geological Prospecting Petroleum Inst.). *Zavod. Lab.*, 1957, **23** (9), 1120–1124. G. S. SMITH

1744. **Derivative thermometric titrations.** S. T. Zenchelsky and P. R. Segatto (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (12), 1856–1858.—The selection of the end-point in a thermometric titration is aided by an apparatus for obtaining first and second derivative curves. Mechanical amplification is used, with a filter to increase signal to noise ratio. K. A. PROCTOR

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British thermal unit	B.Th.U.
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calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	%(v/v)
per cent. (wt. in vol.)	%(w/v)
per cent. (wt. in wt.)	%(w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	$R_{\mathbf{p}}$
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
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volt	V
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watt	W
wavelength	λ
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Volume 5. No. 5, Abstracts 1425-1751 May, 1958

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Printed and Published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, England.
Communications to be addressed to the Editor, Norman Evers, 14, Belgrave Square, London, S.W.1.
Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.
Entered as Second Class at New York, U.S.A., Post Office